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FLUIDIZED BED EVAPORATION OF COAL CAKE CONTAINING PETROLEUM CRUDE OIL

FINAL REPORT

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1.0 EXECUTIVE SUMMARY

1.1 Introduction

This study was undertaken through Supply and Services Canada for the Canada Center for Mineral and Energy Technology (CANMET).

The study investigated the performance of a fluidized evaporator for the evaporation of petroleum crude oil from prepared coal-oil cakes. The experimental fluidized bed evaporator apparatus was provided by CANMET, and the study was performed under the direction of INTEG-Intercontinental Engineering Ltd. in the laboratory and pilot plant facilities of B.C. Research.

1.2 Scope of Work

Under the terms of DSS Contract DSQ81-00027, the study was split into three phases as follows:

Phase I: The apparatus was to be completed and assembled. Coal and oil samples were to be obtained and prepared for testing. Four short tests were to be conducted under predetermined experimental conditions.

Phase II: Five further tests were to be conducted on the feed material prepared in Phase I under experimental conditions provided by CANMET.

Phase III: Additional coal-oil mixture feed material was to be provided by CANMET. Two or three short tests and three to six long tests were to be conducted under experimental conditions provided by CANMET.

In addition to the trial operations, the contract called for analysis of the product samples for oil and water content.

1.3 Summary and Recommendations

A total of sixteen test runs were conducted using a variety of coal-oil cake samples. The experimental apparatus performed reasonably well, and the process appears to be technically possible, with up to 97% of the crude oil content removed from the feed cake.

The effect of process variables on the performance of the process was assessed. The most important process variable was the fluid bed temperature, which exhibited a linear correlation to product oil content. The effects of feed rate, residence time, and feed oil content on product oil content should be investigated further.

Physical properties of the metallurgical coal samples were apparently not adversely affected during trials with windbox temperatures under 470°C. The impact of higher windbox temperatures on coal caking properties should be further explored.

The particulate and condensate collectors proved to be inadequate, and should be improved prior to further trials.

2.0 INTRODUCTION

This report deals with the experimental examination of a Fluidized Bed Evaporator (FBE) designed by the Canada Centre for Mineral and Energy Technology (CANMET) and tested by INTEG-Intercontinental Engineering Ltd. and B.C. Research under Department of Supply and Services (DSS) contract 0SQ 81-00027.

The project was planned and supervised by INTEG staff while the testing was performed in the pilot plant facilities of B.C. Research by INTEG and B.C. Research personnel.

The FBE unit, including cyclone separators and some instrumentation, was borrowed from CANMET and assembled in the B.C. Research pilot area. A mass feeder, solid collectors, condensers, heaters, and additional instruments were purchased by the contractors.

For Phases I and II, metallurgical coal samples were obtained from McIntyre Mines by Saskatchewan Research Council (SRC). These samples were crushed and pulverized under subcontract by Birtley Engineering in Calgary. The pulverized coal was then sent to B.C. Research where it was mixed with Syncrude synthetic crude oil to produce a coal/oil cake containing twelve percent oil by weight.

In Phase III, new sample material was obtained. This material, described in Tables 6.5 to 6.7, consisted of coal/oil centrifuge residues from a parallel project conducted by SRC. The separate samples were combined to give three sets of representative samples representing the three sources - McIntyre, Vesta and Highvale mines, designated as McIntyre, Vesta and Wabamun coals.

The sample crude oil contents were 8.0, 3.6 and 7.2 weight percent, respectively. The McIntyre coal was a metallurgical coal while the

other two were both thermal coals. The crude oil was again a synthetic crude from Syncrude Ltd.

SI units are used throughout this report except in two drawings in Appendix B.

Feed and product samples were collected for laboratory analysis while operating data (pressure, temperature, gas flow, etc.) was collected by the operators during the tests.

3.0 FBE APPARATUS (See Appendices A, B and D)

The FBE apparatus, as described in the Contract, was to be furnished by CANMET. However, the apparatus was not delivered in the condition specified, as some of the equipment was not included. Therefore, additional equipment was purchased and some modifications to the existing apparatus were made.

The delivered equipment included:

(a) FBE body and windbox
(b) FBE support frame
(c) Electric furnace
(d) Cyclone separators with solid collectors
(e) Bed auxiliary heater controller
(f) Rheostats (2) to control FBE body heaters
(g) Thermocouple selector and indicator
(h) Bed solids collector

Equipment purchased and/or modified by the contractors included:

(a) Furnace controller/power supply
(b) FBE body heaters
(c) K-Tron controller and scaffold for installation
(d) Body insulation
(e) Static and differential pressure gauges including all the hardware required for installation.
(f) Thermocouples, wire and fittings
(g) Gas distributor plates
(h) Modifications to K-tron feeder and FBE body to allow vertical discharge required instead of the horizontal arrangement described in the Contract.
(i) Bed product sample collection was enlarged and modified to include a nitrogen purge.

3.1 FBE Body, Cyclones, and Solids Collectors

As previously mentioned, the FBE vessel, two cyclone separators, and three solid product collectors were among the equipment borrowed from CANMET. Initially, these pieces were assembled as received but the bed solids collector and the FBE top were later modified.

3.2 Mass Feeder

A K-Tron LWF-3-35 mass feeder was purchased and installed on a scaffold above the FBE vessel. The feeder outlet was modified on site so that it could be connected to a four inch diameter feed port by a flexible teflon coupling.

The K-Tron controller and power supply were mounted in an air- and dust-tight electrical panel according to the manufacturer's directions. This panel was installed on the FBE support frame where it was easily accessible for set point changes and calibration.

3.3 Nitrogen Supply and Storage

For the first few dry runs with the FBE apparatus, compressed nitrogen in 6 m^3 cylinders supplied the fluidizing medium. The cylinders were connected two at a time to a manifold and the nitrogen was supplied through a standard cylinder regulator and copper tubing.

It was determined, however, that the number of cylinders required and the frequency of replacement made this arrangement impractical. Therefore, it was decided to purchase liquid nitrogen in cylinders which would supply 100 m^3 of gas at standard temperature and pressure. This was sufficient supply for at least two long test runs and the gas supplier was able to deliver a full cylinder within 24 hours of ordering.

The gas delivery system was modified to include a vaporizer and a high flow pressure regulator. The original regulator was moved to a sidestream and used to control the nitrogen purge in the bed solids collector.

3.4 Furnace and Furnace Controller

The primary heat source for the FBE system is a six kilowatt electric furnace through which an Inconel tube is passed. The nitrogen supply flows through the tube, is heated in the furnace and then piped to the FBE windbox.

The furnace itself is electrically connected to a control unit which houses a circuit breaker, temperature indicator and temperature controller. The controller is in turn connected to TI-1, the windbox thermocouple, from which the process temperature is controlled by setting the desired windbox temperature on the controller selector switch.

The furnace and its controller are mounted behind the FBE vessel so that only a short run of insulated tubing is required between the furnace and the windbox.

3.5 Windbox and Distributor Plate

The windbox was included in the equipment borrowed from CANMET. However, a wall heater, thermocouple, pressure tap and a gas distributor plate were added to complete the assembly.

The distributor plate is a sintered, stainless steel disc, 102 mm in diameter and has a free cross-sectional area of 15%. The plate sits in a recess in the windbox flange backed by a perforated steel plate to ensure a sufficient pressure drop across the distributor.

Before bolting the windbox to the FBE body, a combination of ceramic sealant and a gasket was applied to the windbox flange. Without the sealant, it proved impossible to obtain a gas tight joint because the two flanges were not perfectly parallel.

After joining the windbox assembly to the FBE body, the Inconel pipe from the furnace, the thermocouple and the pressure tap were connected to the windbox.

3.6 Exhaust Train

The off-gas (hot nitrogen and petroleum vapours) exits the FBE by means of a 25 mm diameter steel half-coupling which is welded to the top of the FBE unit. The gas is then conveyed to and from the cyclones by 25 mm diameter steel pipe. At the bottom of each cyclone is a short run of 19 mm steel pipe leading to the solid collectors in which the entrained solids are allowed to cool before removal.

From the cyclones, the gas enters the condenser assembly, the construction of which is pictured in Appendix B. The condensers remove some of the evaporated oil and moisture and this condensate is removed by means of the valve at the bottom of each condenser.

A bypass loop permits the isolation of the condenser assembly should the unit become blocked or if it develops a leak.

From the exit of cyclone #2, the gas passes through an orifice meter and then is exhausted through a blower on the roof.

Except for the cyclone and solids collectors, the exhaust train was designed and built by the contractors.

3.7 Auxiliary Heaters and Insulation

In order to provide extra process heat and to prevent condensation of vapours on the walls of the FBE unit, auxiliary electric heaters were installed on the shell of the FBE body and along the off-gas train up to the inlet of condenser #1.

At full power, these heaters are rated at 5.7 kW but are usually under the control of a temperature controller (bed area) or two rheostats.

The heated areas are also covered by at least 25 mm of ceramic fibre insulation.

3.8 Instrumentation and Controls (See Appendix A)

The FBE unit was equipped with the necessary devices for the monitoring and control of important process variables.

Type K thermocouples were installed to measure the temperature of the process nitrogen, interior and exterior FBE temperatures and the off-gas temperature. The mid-bed and windbox thermocouples are also connected to the bed heater and furnace control loops.

Pressure taps, equipped with filter/snubber assemblies were connected in order to measure nitrogen delivery pressure, distributor plate differential pressure (DP), bed DP, cyclone DP, off-gas orifice DP and off-gas pressure. The nitrogen pressure, orifice DP and off-gas pressure were used to calculate gas flows in the FBE, the bed DP was correlated to approximate bed height and the cyclone DP indicated the need to empty the solids collectors or clear a block in the cyclones.

The nitrogen flows in the FBE were controlled by the nitrogen pressure regulator, PR-1, and the flow control valve, CV-1. The regulator was

set at 345 kPa(g) throughout the tests while CV-1 was adjusted to deliver the desired flow (normally 85 L/min). The gas flows are measured by a rotameter on the inlet nitrogen stream and an orifice meter, FE-1, at the flue gas outlet.

As previously mentioned, the coal/oil cake feed rate was regulated by the K-Tron control unit. The K-Tron is designed as a loss-in-weight feeder, however, it was discovered that it operated better in the volume mode at the relatively low feed rates required for the experiments.

4.0 OPERATING PROCEDURES

4.1 Preliminary Routine

4.1.1 K-Tron Calibration

Prior to each set of tests, the K-Tron mass feeder was calibrated according to its instruction manual. Briefly, this procedure entails removing the FBE windbox, collecting the feed material in a tared container over a measured time interval, calculating the feed rate and comparing it to the controller set point. If the two rates do not match, the necessary adjustments are made in the controller and the procedure is repeated.

4.1.2 Leak Tests

After FBE equipment was completely assembled, the nitrogen was turned on to a rate and pressure above normal operating conditions. Using some "SNOOP", a liquid leak detector, gas leaks were located and sealed.

4.1.3 Exhaust System Inspection

Before each test, the exhaust system was checked for clear passage, the roof blower turned on and the condenser cooling water turned on.

4.2 Startup Routine

4.2.1 Nitrogen Flow

During the initial startup period, the nitrogen flow was set to 30% on the rotameter (approx. 25 L/min).

Upon reaching the desired operating temperature, the nitrogen flow is increased to 65% (85 L/min).

4.2.2 Furnace and Wall Heaters

The heaters and furnace were adjusted to the following settings for the startup period:

(a) Rheostat #1 (windbox heater, freeboard wall heaters) - 80 to 100%.
(b) Rheostat #2 (Upper bed area, exhaust train) - 25 to 50%.
(c) Auxiliary heaters on upper bed and freeboard walls - ON.
(d) Bed temperature controller - required temperature.
(e) Furnace - Windbox temperature required to maintain bed temperature (400 to 475°C).

The FBE unit normally required about three hours to reach operating temperature.

4.2.3 K-Tron Mass Feeder

While the FBE is heating, the K-Tron mass feeder is filled and sealed, the feed rate set, the display switch set to MASS and the delivered mass totalizer zeroed. If temperatures and nitrogen flows are at or near operating range, the feeder was normally run for a few minutes to check for proper operation.

4.2.4 Cyclones, Condensers, and Collectors

As the FBE unit approaches operating temperature, the condensers, cyclones and collectors are cleared of any old material and clean sample containers are prepared.

4.3 Procedures at Operating Conditions

4.3.1 K-Tron Mass Feeder

When the FBE unit is at the desired temperature and nitrogen flow, the K-Tron may be switched on to begin feeding (Mode switch to VOL). The feeder should now be displaying and delivering the set feed rate.

4.3.2 Product Sampling and Bed Level Control

The bed material may be removed for sampling and/or level control in two ways:

(a) Open the sample valve to set up a continuous flow of product material equal to the coal cake feed rate.
(b) Remove material frequently in small batches.

Method (a) has proved impractical except at feed rates greater than 15 kg/h. In both cases, coal product should be removed at such a rate so as to maintain the bed differential pressure between 50 and 100 mm of water column. This measurement is equivalent to 35 to 45 centimeters of bed height, the range in which the FBE operation appeared to be most stable.

The cyclone collectors should also be emptied periodically to prevent plugging of the cyclones. The cyclone normally ran at 125 to 250 mm of water column and an increase to above 250 mm usually indicated the formation of a block somewhere in the cyclones. These plugs normally began at the solids outlet of the cyclone and were easily cleared by poking a steel rod into the cyclone.

The solid product removed from the bed and cyclones was stored under nitrogen until cool and was later weighed to determine the average feed rate of the test.

4.3.3 Condensate Sampling

The liquid condensate was removed frequently throughout a test run because entrained solids carried over from the cyclones tended to settle in the condensers (especially #1) and plug the condensate drain.

When sampling, the condensate was drained from both condensers into a tared container and at the end of a run the composite sample was weighed. The sample was then mixed thoroughly and a representative sample taken for later analysis.

4.3.4 Data Acquisition

Operating data was collected manually during the test runs. Readings of temperatures, pressures, etc. were recorded every thirty minutes or less. The operators also kept a log book in which observations were noted.

4.4 Shutdown Procedure

When a test run was completed, the K-Tron, heaters and furnace were all shut off. The nitrogen flow was reduced to 30% (25 L/min).

After the FBE had cooled for two to three hours, the nitrogen and condenser cooling water were turned off.

5.0 ANALYTICAL METHODS

5.1 Phase I and II Analyses

5.1.1 TGA Procedure for McIntyre Metallurgical Coal

For Phases I and II, the feed material and products were analyzed for oil content by thermogravimetric analysis (TGA) on a Dupont 951 thermogravimetric analyzer.

Samples of feed and product coals were coned and quartered to yield representative samples of about 50 milligrams each. This sample was then placed on the microbalance of the analyzer and heated to 120°C in a nitrogen atmosphere. The weight loss was recorded and used to estimate the weight percent of moisture in the sample.

The sample temperature was then raised and held at 460°C where the corresponding weight loss was used to determine the oil concentration in the coal cake.

After several minutes at 460°C, the temperature was increased to 900°C giving the percentage of coal volatiles.

Oxygen was then introduced into the sample cell, the sample burned, and the percentages of fixed carbon and ash were calculated.

Examples of Phase I and II TGA recordings are included in Appendix C. It should be noted that the solid trace represents the sample weight loss while the dashed line shows the temperature program.

5.1.2 Random Errors

For each run, A through D in Phase I, one product sample was taken 30 to 60 minutes before the end of the tests and another was taken at the end of the runs. Table 5.1 shows that the measured oil contents were in good agreement for a given test and the largest error observed was 0.5 % wt.

Therefore, random errors on oil contents determined by TGA were limited to 0.5% wt and include both analytical and sampling errors.

5.1.3 Systematic Errors in TGA Analyses

Table 5.2 shows that 0.15% of the crude oil remains unvaporized at 460°C. Assuming there is no interference of coal on oil evaporation, the 0.15% of oil content of the feed and product coals is unaccounted for by TGA. This may result in the following systematic errors:

(a) Feed at 12% wt oil -
Error = 12% x 0.0015 = 0.02% wt

(b) Product at 2.2% wt oil -
Error = 2.2% x 0.0015 = 0.003% wt

Again in Table 5.2 it can be seen that the moisture content of the McIntyre coal used in Phases I and II was 0.5% wt. This water is accounted for when calculating the oil content of the feed coal cake but assumed to be zero in the products. If, in fact, none of this moisture is removed by the FBE, then the 0.5% wt represents the maximum systematic error attributable to unevaporated coal moisture in the product coals.

Analyses also showed a possible weight loss of up to 0.2% wt from the pure coal between 120°C and 460°C (not shown in Table 5.2). This must also be considered as a systematic error in the TGA's of feed and product materials.

5.1.4 Caking Properties of Phase I Coal

At the request of the Scientific Authority, additional tests were performed on the Phase I raw coal and on the products from Runs A through D inclusive. These tests were to determine whether FBE processing had affected the caking properties of the McIntyre metallurgical coal.

These tests were performed by General Testing Laboratories, Vancouver, B.C. and included:

(a) Free swelling index of coal - ASTM D720.
(b) Plastic Properties of Coal by the Constant Torque Giesler Plastometer - ASTM D2639.
(c) Determination of the Degree of Coking of Bituminous Coal by the Dilatomer Method - DIN 51739

5.2 Phase III Analyses

5.2.1 TGA of Vesta and Wabamun Coal Cakes

The Vesta and Wabamun thermal coals and their coal/oil cakes were analyzed by the TGA method described in Section 5.1.1.

However, this method could not be used to determine the residual oil in the product coal because both oil and coal volatiles were evaporated at temperatures between 120°C and 460°C. The product residual oil was determined by Soxhlet extraction.

5.2.2 Oil Contents by Soxhlet Extraction

Residual oil in the feed and product coals was extracted by refluxing with hexane for sixteen hours in a standard Soxhlet extraction apparatus. Hexane was the chosen solvent because it extracted the oil completely but did not remove any significant amount of the coal. Toluene, for example, would have dissolved four to five weight percent of the coal.

The extracted oil/hexane mixture was then transferred to a rotary evaporator where most of the hexane was removed. The evaporator flask, containing mainly oil, was placed in a vacuum drying oven and the residual hexane evaporated.

Errors involved in this method arose mainly from weighing the evaporator flask and the flask plus oil. For each weight measurement, there was an error of $\pm$ 0.05 grams for a total error of $\pm$ 0.1 grams. In the case of the feed coals, the sample weight was 20 grams resulting in an oil content error of:

$$(\pm 0.1g \div 20g) \times 100 = \pm 0.5\%$$

Similarly, the error in residual oil content of the product coals was $\pm$ 0.1% to $\pm$ 0.2% given sample sizes of 50 to 80 grams.

5.2.3 Moisture Analysis

Since there was some question about the validity of the moisture contents from thermogravimetric analysis, the Vesta and Wabamun moistures were determined by co-distillation with xylene.

5.2.4 Analysis of Phase III McIntyre Coal

The McIntyre feed and product mixtures were similar to that used in Phases I and II. Therefore, the same TGA method was applied in determining the oil and moisture contents.

TABLE 5.1

Phase I Product Oil Content Versus Elapsed Time

Test Run	A	B	C	D
Elapsed Time, min.	20	88	83	59
Product Oil Content, % wt	1.4	1.6	1.9	2.2
Elapsed Time, min.	45	118	113	104
Product Oil Content, % wt	1.9	1.4	2.2	1.7
Elapsed Time, min.	217	-	-	-
Product Oil Content, % wt	1.3	-	-	-
Elapsed Time, min.	217	-	-	-
Product Oil Content, % wt	1.4	-	-	-

TABLE 5.2

Phase I and II TGA – Determination of Systematic Errors

Sample	Pure Coal #3 (1)	Crude Oil (2)	Feed Coal #1 (3)	Product Coal BC 15 (4)
% Wt. loss below 120°C	0.5	38.2	4.9	0.1
% Wt. loss between 120°C and 460°C	0	61.7	7.7	1.2
% Wt. loss over 460°C	17.3	0.15	15.0	16.7
% Fixed carbon	74.7	-	65.9	75.6
% Ash	7.5	-	6.5	6.5
% (Oil + Moisture)	0.5	99.9	12.6	1.3
% Oil	0	100	12.0	-

Notes:

(1) Pure Coal #3 = one of the grab samples of pulverized but unmixed McIntyre coal used in Phases I and II.

(2) Crude Oil = Syncrude synthetic crude oil as received by the contractors.

(3) Feed Coal #1 = grab sample of the 12% wt oil in coal feedstock prepared for Phases I and II.

(4) Product Coal 15 = a product coal grab sample taken during Phase I, Run A.

6.0 TEST RESULTS

6.1 Operating Problems and Solutions

6.1.1 Fluidization and Solids Flow

During the Phase I shakedown tests, problems were experienced in bed fluidization and solids flow within the FBE vessel.

The first problem area identified was the vertical partition installed in the FBE. The partition was designed to increase the residence time of the coal cake in the FBE but only succeeded in causing plugs at the transition between the freeboard area and the four inch diameter bed section.

After removing the partition, bridging still occurred in the four inch section at the nitrogen flows specified in the original Phase I test conditions (Table 6.1). Therefore, a series of short tests were performed in order to determine the minimum nitrogen flow required to fluidize the bed. This flow rate was found to be about 80 L/min (at 20°C, 100 kPa) and flows of 80-90 L/min were used for the remaining runs.

6.1.2 Solids Entrainment

The increased nitrogen flow rate resulted in a significant increase in solids entrainment in the off-gas. Measurements made in Phase I indicated that some 8 or 9% of the coal cake fed was removed from the cyclones plus an unknown quantity dropping out in the condensers.

The amount of entrained coal particles was sufficient to plug the entrance of condenser #1 on a few occasions although a constriction at the inlet was partly to blame for this. Fortunately, this area was readily accessible for preventive and emergency maintenance.

As previously mentioned, the high particulate loading required that the cyclone collectors be cleared frequently in order to prevent plugging at the bottom outlet of the cyclones.

6.1.3 Gas Leaks

The most irritating problem for the FBE operators was the persistent gas leakage from the system.

The worst areas were the flanges on the FBE body itself (windbox, freeboard/bed area junction and FBE top), and the tops of the two condensers. All were "homemade" flanges, made from short metal, without a proper sealing face and most were warped to the point where gaskets alone were of little use.

In all cases, it was necessary to use some combination of gaskets, high temperature ceramic sealant and silicone sealant in order to obtain an effective, gas-tight joint. Before each run, all joints were inspected for leaks which occasionally resulted from thermal expansion of the apparatus.

6.2 Results of Analyses

6.2.1 Phase I and II Results

Test runs A through J were performed using the McIntyre metallurgical coal containing 12% wt synthetic crude oil. From the product weights, TGA analyses, temperatures and pressures, the feed rates, FBE temperatures and nitrogen flows were calculated. These results are presented in Table 6.3.

The results of the analyses of caking properties for the raw McIntyre coal and the products of tests A to D are presented in Table 6.4.

During the testing program, the effects of demister elements on condenser efficiency was tested. Although these experiments were conducted during all phases of testing, all of the results are presented in this section. These figures are recorded in Table 6.5.

6.2.2 Phase III Test Results

In Phase III, a series of centrifuge underflow samples were collected and forwarded by SRC to use as feedstock for the FBE. Tables 6.6 to 6.8 present the characteristics of the various samples.

From the fourteen samples, three feed mixtures were prepared as follows:

(a) McIntyre - combination of MC-2 to MC-5.
(b) Vesta - combination of V-1, V-2, V-4 and V-5.
(c) Wabamun - combination of W-1 to W-4.

The McIntyre mixture was used in test K, the Vesta in tests L, P and Q while the Wabamun was feed for tests M, N and O. The characteristics of the three feeds are shown in Table 6.9 along with the other results of Phase III testing.

6.3 Carbonization, Combustion, Crude Oil and Condensate Evaluation Tests

CANMET will conduct tests for evaluation of coking and combustion properties of orginal, pulverized, metallurgial and thermal coals and coal products as well as crude oil and condensate samples.

TABLE 6.1

Predicted Phase I Test Conditions

Test Run	A	B	C	D
Feed Rate, kg/h	2.3	4.5	7.4	14.2
Windbox Temp., °C	300	400	300	400
Bed Temp., °C	230	250	220	240
Nitrogen Flow, L/min (20°C, 100 kPa)	22.2	20.8	66.4	61.9
Product Oil Content, % wt	1.0	1.5	2.5	3.0

Note: Feed cake oil content = 12% wt

TABLE 6.2

Phase I and II Test Results

Test Run	A	B	C	D	E	F	G	H	J
Feed Rate, kg/h	2.3	4.6	8.5	17.2	5.2	15.3	4.7	6.7	11.3
Windbox Temp., °C	367	396	427	472	408	459	414	452	469
Bed Temp., °C	287	291	239	252	269	239	267	265	264
Freeboard Temp., °C	286	290	251	238	252	222	261	258	256
Shell Temp., °C	-	-	-	-	-	-	230	238	227
Nitrogen Flow, L/min (20°C, 100 kPa)	76.2	89.3	87.5	90.6	82.1	86.3	85.4	84.7	88.3
Flue Gas Flow, L/min (20°C, 100 kPa)	81	89	87	89	87	-	105	95	85
Product Oil Content, % wt	1.3	1.4	2.2	1.7	1.4	2.1	1.4	1.5	1.4

Note: (1) Feed Oil Content = 12% wt
(2) Tests A to D - readings are those at equilibrium.
(3) Tests E to J - reasings are averages for the test.
(4) Freeboard temperature is average of TI-4 to TI-6.

TABLE 6.3

Phase I: Results of Caking Property Analyses

Test Run	Raw Coal	A	B	C	D
Softening Temp., °C	434	431	428	428	446
Max. Contraction, Temp. °C	509	503	515	509	512
Contraction, %	19	20	20	23	8
Dilation, %	Nil	Nil	Nil	Nil	Nil
Max. Fluidity, dd/m	Nil	Nil	Nil	Nil	Nil
Free Swelling Index	6	4.5	5	4.5	3.0 & 3.5

TABLE 6.4

Condenser Efficiency

Test Run	Theoretical Condensate Yield, kg	Actual Condensate Yield, kg	Condenser Efficiency %
Preliminary Test (1)	1.8	0.64	36
Preliminary Test	0.4	0.05	13
A (2)	0.8	0.14	18
B	0.9	0.13	14
E (3)	1.3	0.98	75
G	2.6	1.6	62
H	2.8	1.4	50
K	3.0	3.2	107
L	7.7	2.6	34
M	3.5	2.1	60
N	1.7	0.74	44
O	0.59	0.30	51
P	3.3	1.3	39
Q	1.4	1.4	52

Notes: (1) No demister element in preliminary tests.
(2) Tests A & B - 6 layers of wire mesh for demister element.
(3) Tests E to Q - 12 layers of wire mesh.

TABLE 6.5
Phase III McIntyre Coal: Oil Content of Centrifuge Underflow Samples

Sample	Centrifuge Feed Rate L/min	Oil Content % wt
MC-1	0.5	6.7
MC-2	0.5/1.0	6.9
MC-3	1	6.8
MC-4	4	9.1
MC-5	7.5	10.5

TABLE 6.6
Vesta Coal: Oil Content of Centrifuge Underflow Samples

Sample	Centrifuge Feed Rate L/min	Oil Content % wt
V-1	1	3.2
V-2	4	4.2
V-3	0.5	2.0
V-4	7.5	3.4
V-5	Composite	3.6

TABLE 6.7
Wabamun Coal: Oil Content of Centrifuge Underflow Samples

Sample	Centrifuge Feed Rate L/min	Oil Content % wt
W-1	0.5	5.7
W-2	4	5.8
W-3	7.5	5.9
W-4	Composite	6.4

TABLE 6.8

Phase III Test Results

Test Run	K	L	M	N	O	P	Q
Coal Type	McIntyre	Vesta	Wabamun	Wabamun	Wabamun	Vesta	Vesta
Feed Oil Content, % wt	8.0	3.6	7.2	7.2	7.2	3.6	3.6
Feed Moisture Content, % wt	0.5	19.2	12.1	12.1	12.1	19.2	19.2
K-Tron Setpoint, kg/h	11.0	5.0	7.0	5.0	3.5	5.0	3.5
Actual Feed Rate, kg/h	12.7	9.7	9.1	8.1	3.6	9.7	6.6
Windbox Temp., ^{o}C	465	456	458	433	414	436	425
Bed Temp., ^{o}C	224	144	156	208	237	162	194
Shell Temp., ^{o}C	218	150	176	203	240	170	205
Nitrogen Flow, L/min	86.0	90.3	85.5	87.4	87.4	87.4	87.8
Product Oil Content, % wt	3.1	1.6	3.4	0.8	0.2	1.3	0.7
Product Moisture Content, % wt		0	2.0	1.6	0.8	0.4	0.6

7.0 DISCUSSION OF RESULTS

7.1 Phase I and II Results

7.1.1 Effects of Bed Temperature

Figure 7.1 shows that the FBE bed temperature had a major effect on the oil content of the product coal. A statistical analysis resulted in a linear regression correlation coefficient of $r = -0.88$. This means that over 77% of the product oil content variation is accounted for by its linear association with the FBE bed temperature.

By extrapolating the curve, it can be predicted that a 0% oil content could be achieved at a bed temperature of about 360°C (given the other process conditions are within the ranges used in Phases I and II). This figure compares favourably with that of 350°C derived from the crude oil TGA in Appendix C.

7.1.2 Effects of Other Process Variables

Variations of product oil content and oil evaporation rate with variables such as coal cake feed rate, windbox temperature and differential between windbox and bed temperature of runs of Phase I and II are summarized in Table 7.1.

Correlations of product oil content with coal cake feed rate, Figure 7.2, and windbox temperature, Figure 7.3, resulted in coefficients of determination of 0.58 and 0.23, respectively. Obviously, a detailed study of the effect of these two variables on the product oil content would be required. However, such a study was beyond the scope of this contract.

A cubic polynominal regression of the oil evaporation rate with the differential of windbox and bed temperature is shown in Figure 7.4. Apparently, a distinctively faster and required oil evaporation, larger than 0.5 kg/hr, occurred within the temperature differential between 160 and 180°C. It implies that case of heat supply, exclusively by fluidization gases, the windbox temperature should be as high as possible. However, as discussed in the next section, 7.1.3, there would be some limits for the upper level of this temperature.

7.1.3 Effect of Processing on Coal Caking Properties

The coking properties of the pure McIntyre coal and the coal products from tests A through D were analyzed by various methods (See Sections 5.2 and 6.2.1 as well as Table 6.4). The products of runs A to C were relatively unchanged from the pure coal but run D exhibited a marked difference in free swelling index and maximum contraction.

If the results in Table 6.3 are examined, the only major difference in operating conditions appears to be the windbox temperature. Apparently, the coal was exposed to these higher temperatures in the distributor plate area and this may have changed the coking properties of the coal. However, further experiments are required to verify this observation.

7.2 Condenser Efficiency

The results presented in Table 6.5 show that the crude demister element installed in condenser #2 made a significant difference in condensate recovery. This indicates that one of the commercially available mesh entrainment separators (Otto York Co., or Armstrong-Jones Ltd.) might be an effective method of removing the condensate from the flue gas stream.

7.3 Phase III Results

7.3.1 McIntyre Coal Sample

One test was performed using the McIntyre composite containing eight percent crude oil. Although one test cannot be considered conclusive, it appeared to follow the trends set by Phase I and II runs.

7.3.2 Volatile Losses From Vesta and Wabamun Coals

Thermogravimetric analyses of the Vesta and Wabamun thermal coals revealed that a large fraction (Vesta, 32% and Wabamun, 27%) of both coals was volatilized before 460°C. Of this fraction, about half was moisture (see Table 6.9) and the rest was light volatiles.

The effect of these volatiles and moisture was twofold:

1. the increased evaporative load limited the performance of the FBE and

2. the light volatiles prevented the use of TGA in determining the oil content of the product coal.

The original testing program was modified in that feed rates and bed temperatures were lower while the TGA analyses was replaced by Soxhlet extractions and xylene co-distillation (see Section 5.3).

7.3.3 Effects of Bed Temperature

Figure 7.5 shows that there is a strong linear relationship between the FBE bed temperature and the product oil content for both the Vesta and Wabamun coals. A similar relationship was found for the McIntyre coal in Phases I and II.

TABLE 7.1

FBE Bed Heat Input and Oil Evaporation Rate

Run	Feedrate kg/h	Feed Oil Content % wt	Product Oil Content % wt	Oil Evaporation Rate kg/h	Windbox Temp. °C	(Windbox Temp.) - (Bed Temp.) °C
A	2.3	12	1.3	0.25	367	80
B	4.6	12	1.4	0.55	396	105
C	8.5	12	2.2	0.83	427	188
D	17.2	12	1.7	1.77	472	220
E	5.2	12	1.4	0.55	408	139
F	15.3	12	2.1	1.51	459	220
G	4.7	12	1.4	0.50	414	147
H	6.7	12	1.5	0.70	452	187
I	11.3	12	1.4	1.20	469	205

7.3.4 Effects of Other Variables

Figure 7.6 shows plots of the oil and water evaporation rate versus the differential between windbox and bed temperatures for Vesta and Wabamun coal-oil mixtures. Lack of more data should account for a relatively poor correlation. Effect of other variables on the evaporation of thermal coal-oil mixtures may probably be complicated by the simultaneous evaporation of large amounts of water. Similarly, as in the case of metallurgical coal, a detailed exploration study was beyond the scope of this contract.

A distinctive difference in the volatile matter evaporation of Wabamun, Vesta and McIntyre coal-oil mixutres is obvious from comparison of slopes of curve, -0.016, -0.018 and -0.041, respectively, in Figures 7.1 and 7.5.

7.3.5 K-Tron Instability

In tests L through Q, the K-Tron mass feeder varied from its set point by as much as +94% (eg. tests L and P). This occurred in spite of the fact that the feeder was calibrated before test L and it appeared to be operating correctly in test K. Throughout tests L to Q, the controller was displaying feed rates very close to the set point.

No solution to this problem was discovered at the time of preparing this report.

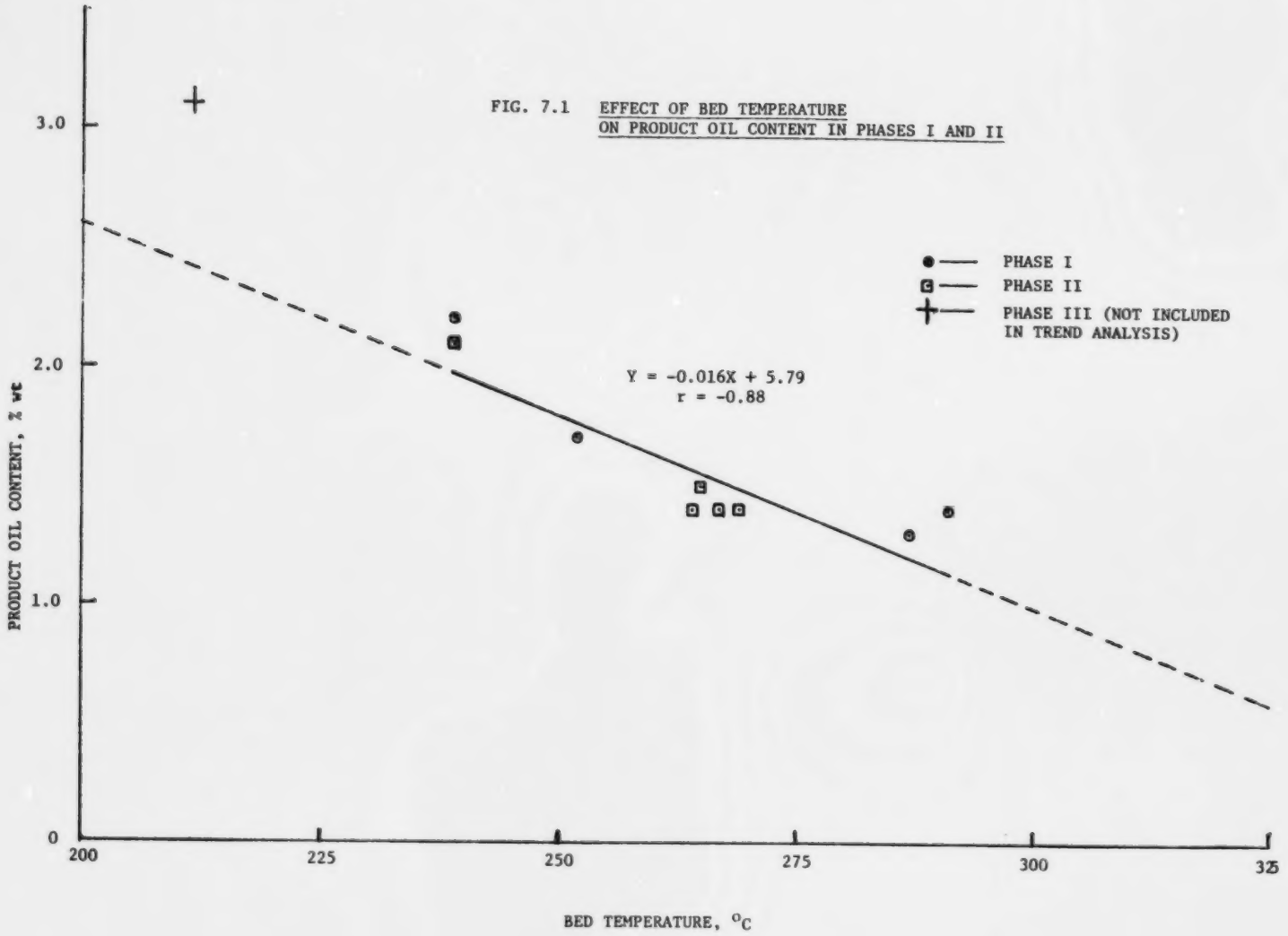


FIG. 7.1 EFFECT OF BED TEMPERATURE ON PRODUCT OIL CONTENT IN PHASES I AND II

FIG. 7.2 PHASE I & II: PRODUCT OIL CONTENT VERSUS COAL CAKE FEED RATE

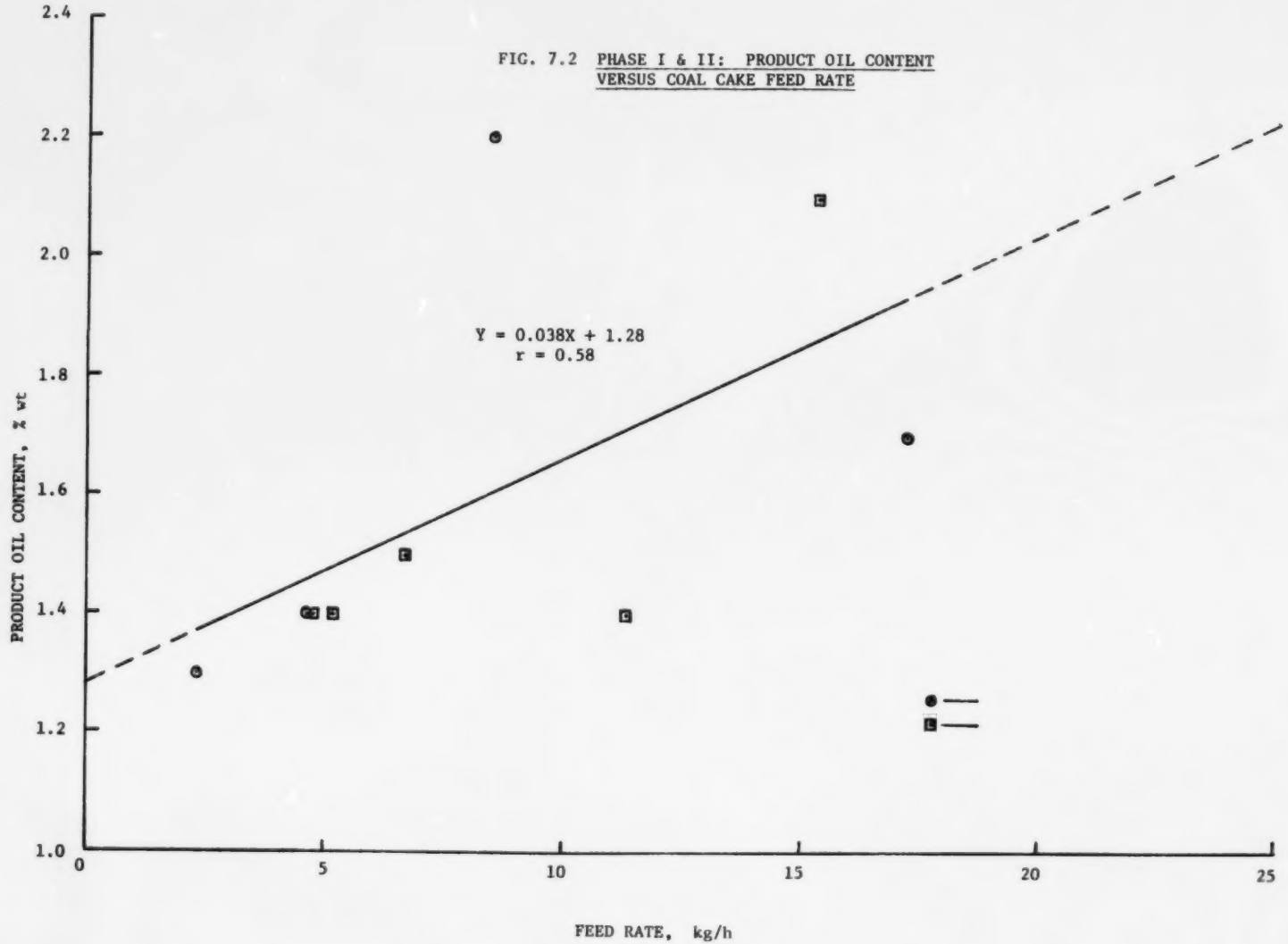


FIG. 7.3 PHASES I & II:
EFFECT OF WINDBOX TEMPERATURE
ON PRODUCT OIL CONTENT

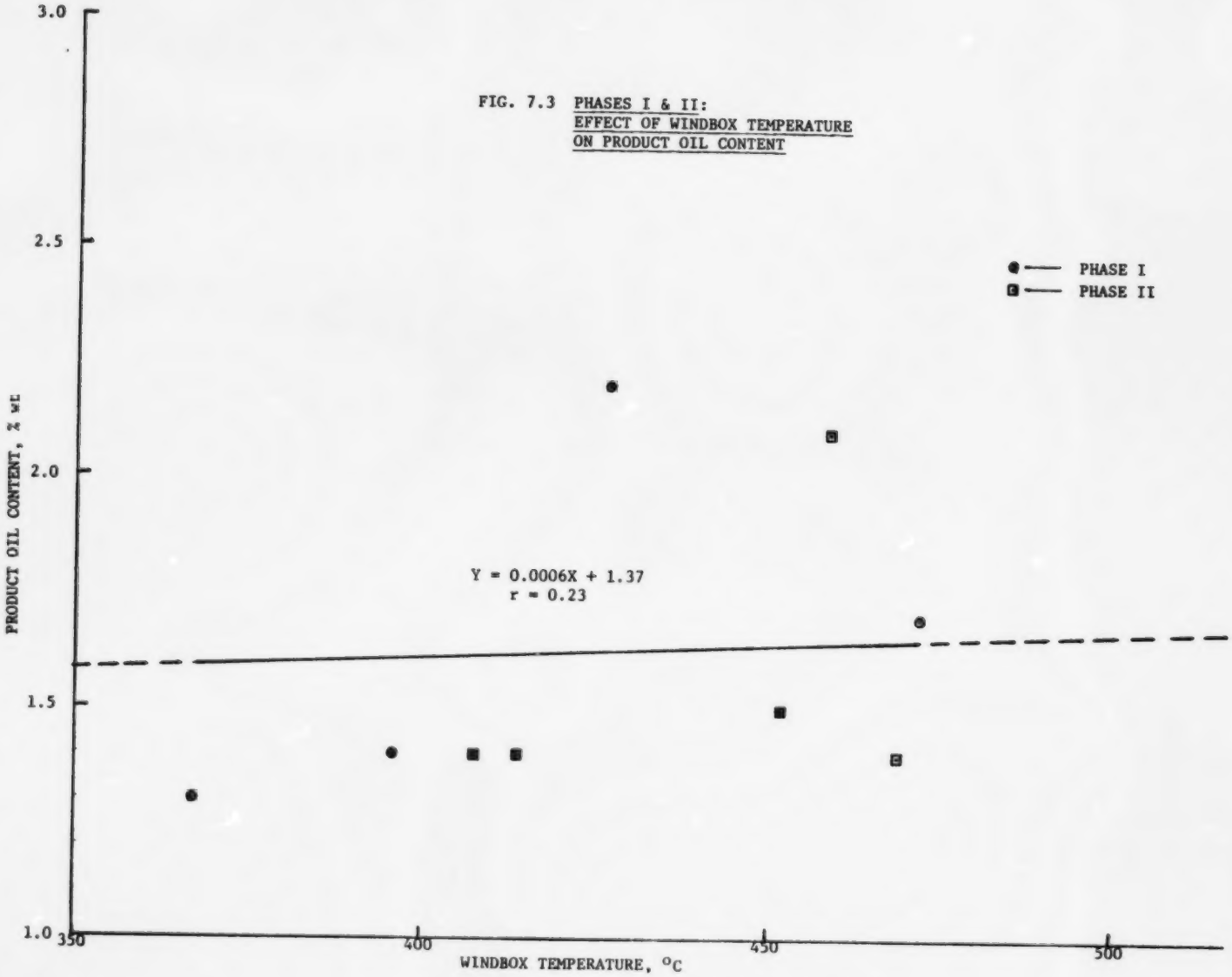
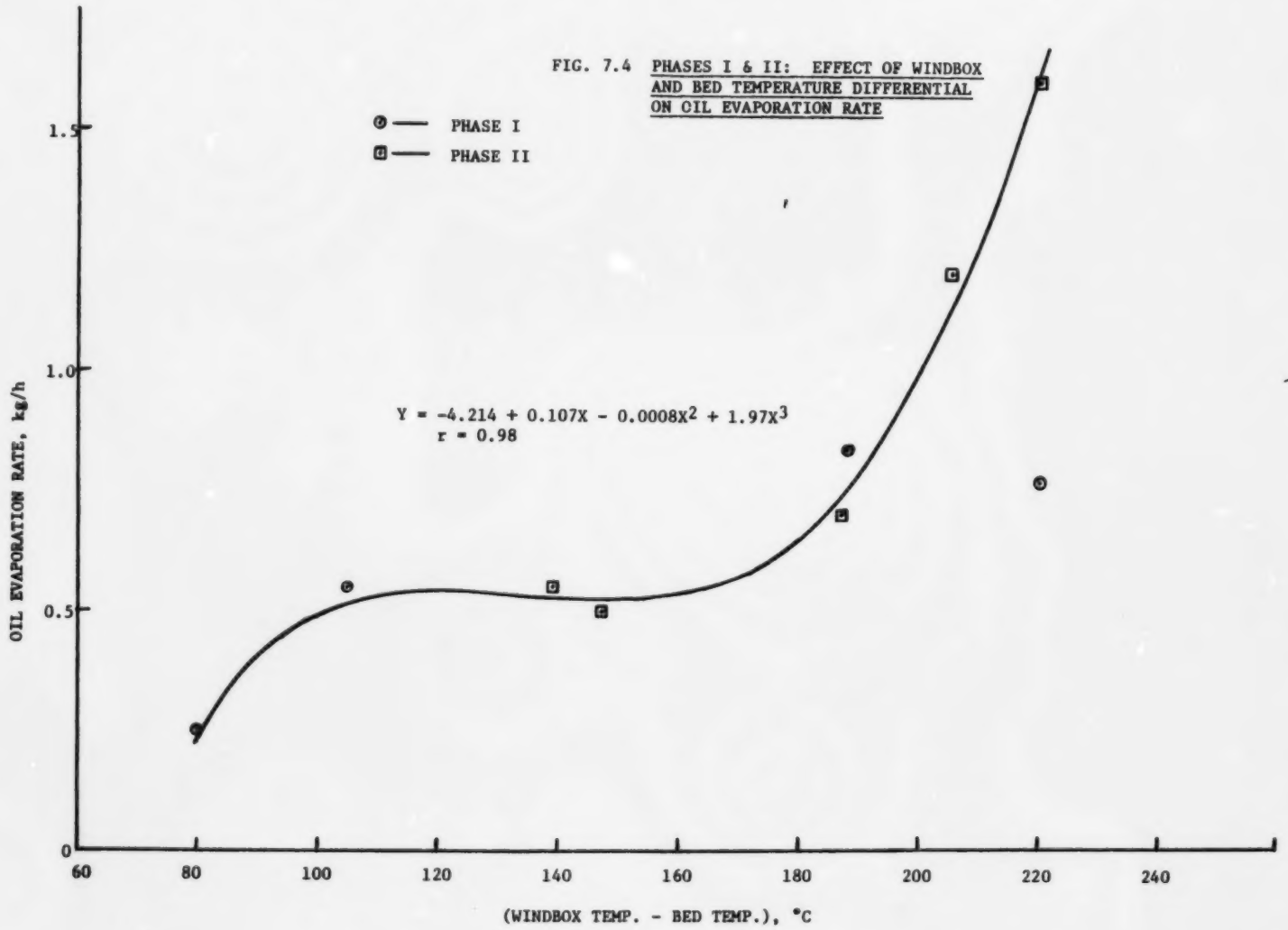


FIG. 7.4 PHASES I & II: EFFECT OF WINDBOX AND BED TEMPERATURE DIFFERENTIAL ON OIL EVAPORATION RATE



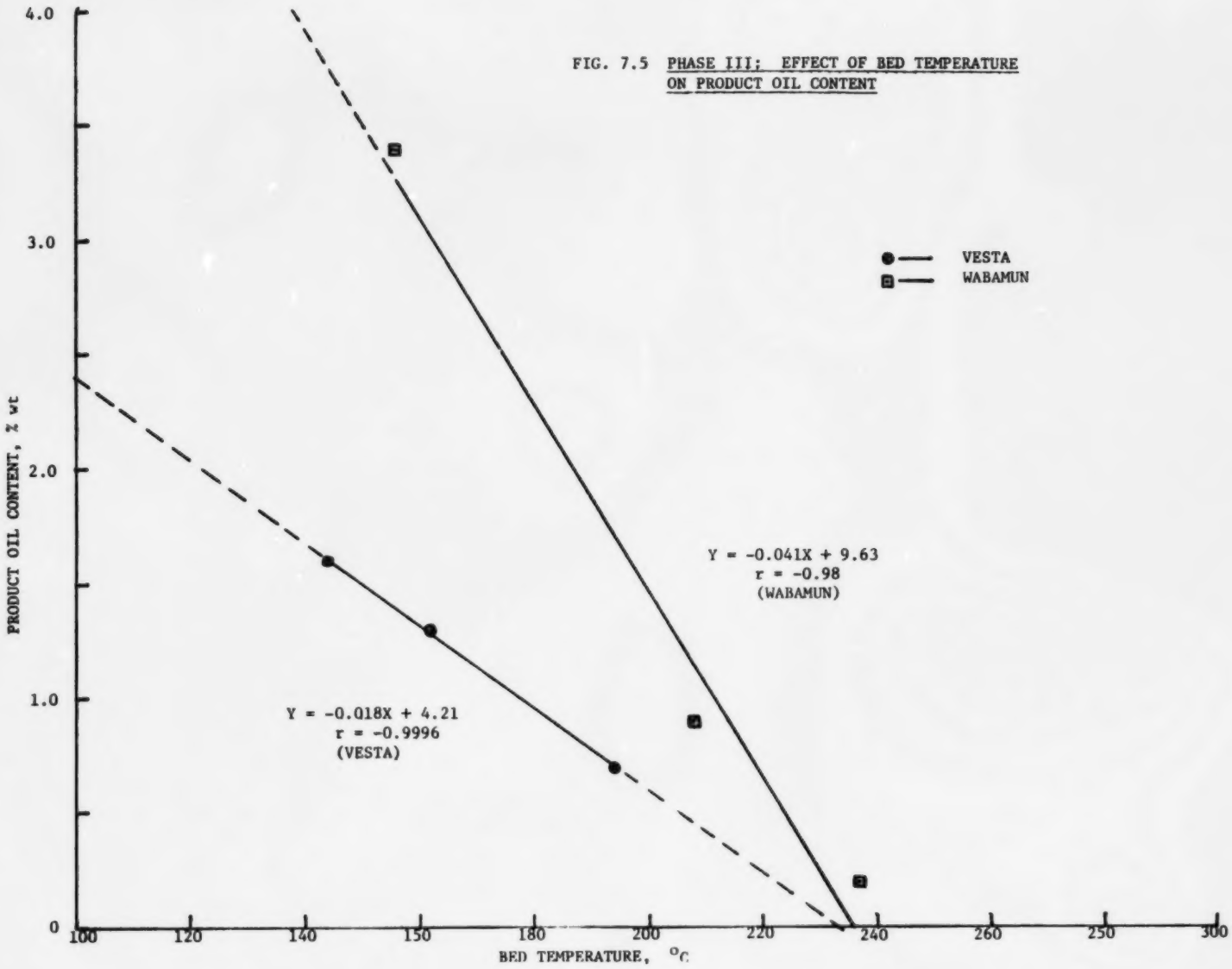
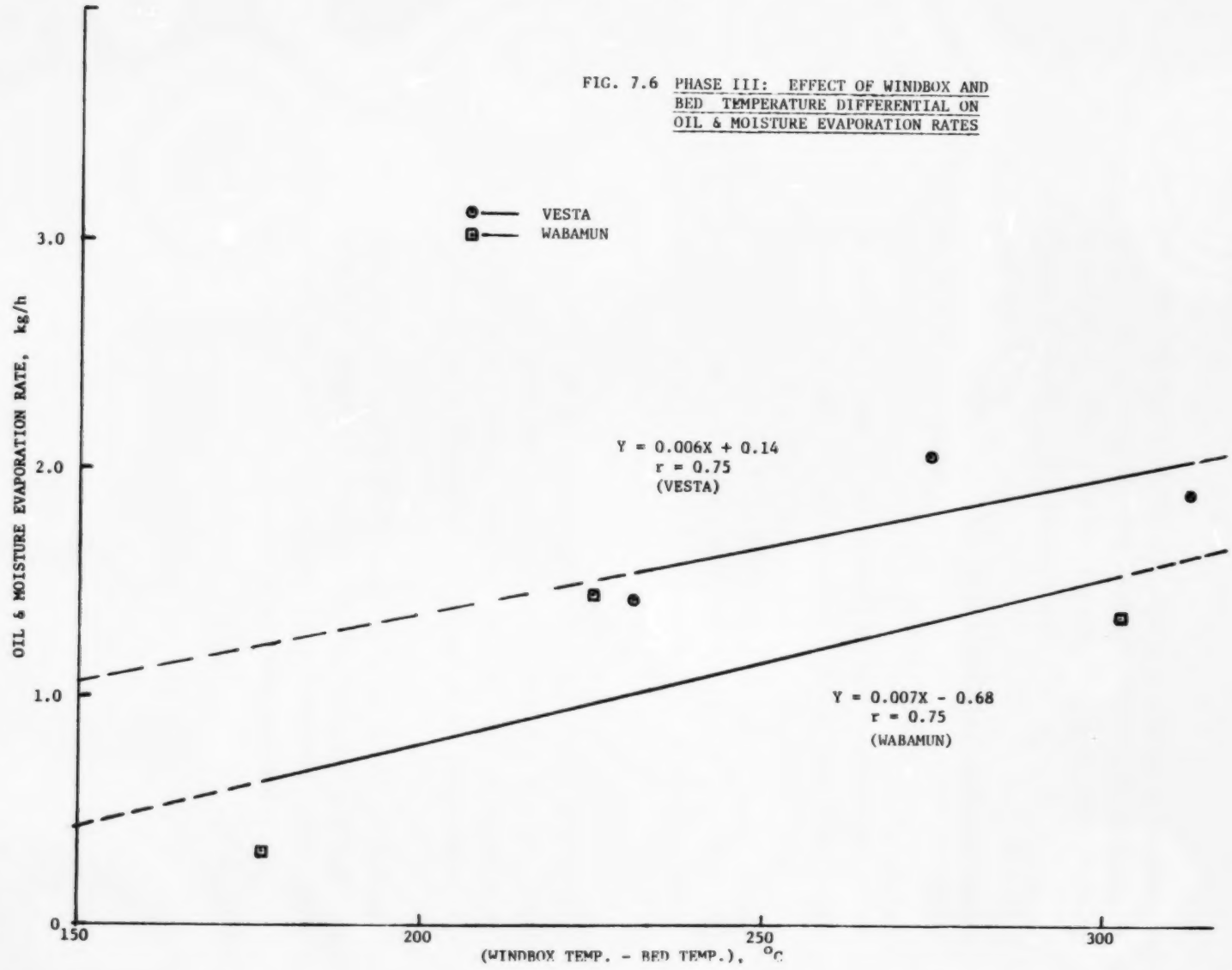


FIG. 7.5 PHASE III; EFFECT OF BED TEMPERATURE ON PRODUCT OIL CONTENT

FIG. 7.6 PHASE III: EFFECT OF WINDBOX AND BED TEMPERATURE DIFFERENTIAL ON OIL & MOISTURE EVAPORATION RATES



8.0 CONCLUSIONS

(a) The FBE process is technically feasible. However, coking and combustion related properties of coal products will still have to be evaluated. During the testing program, up to 97% of the oil content of the feed cake was removed.

(b) At windbox temperatures less than 470°C, the caking properties of the McIntyre metallurgical coal were not adversely affected.

(c) As expected, residue oil content in coal products (in % wt) decreases linearly with increasing bed temperature (°C). Slopes with empirical correlations are -0.16, -0.18, and -0.041 for McIntyre, Vesta, and Wabanum coals, respectively. Extrapdated maximum bed temperature (0 % wt residue oil content in coal product) is approximately 360°C for McIntyre coal and 235°C for Vesta and Wabamun coals.

(d) Improvement of the exhaust gas solid separators is indicated. The high particulate load in the off-gas resulted in occassional plugging of the cyclone collectors and the entrance to condenser #1. Some coal particles were also present in the condensate.

(e) Tests indicate a mesh entrainment separator may be an effective method of condensate recovery. Demister elements installed in condenser #2 resulted in a significant improvement in condensate recovery over test runs in which no demister was used.

9.0 RECOMMENDATIONS

(a) The effect of windbox and bed temperature on the physical properties of coal should examined in greater depth. Tests in Phase I indicated the caking properties of the coal are affected at windbox temperatures higher than 470°C.

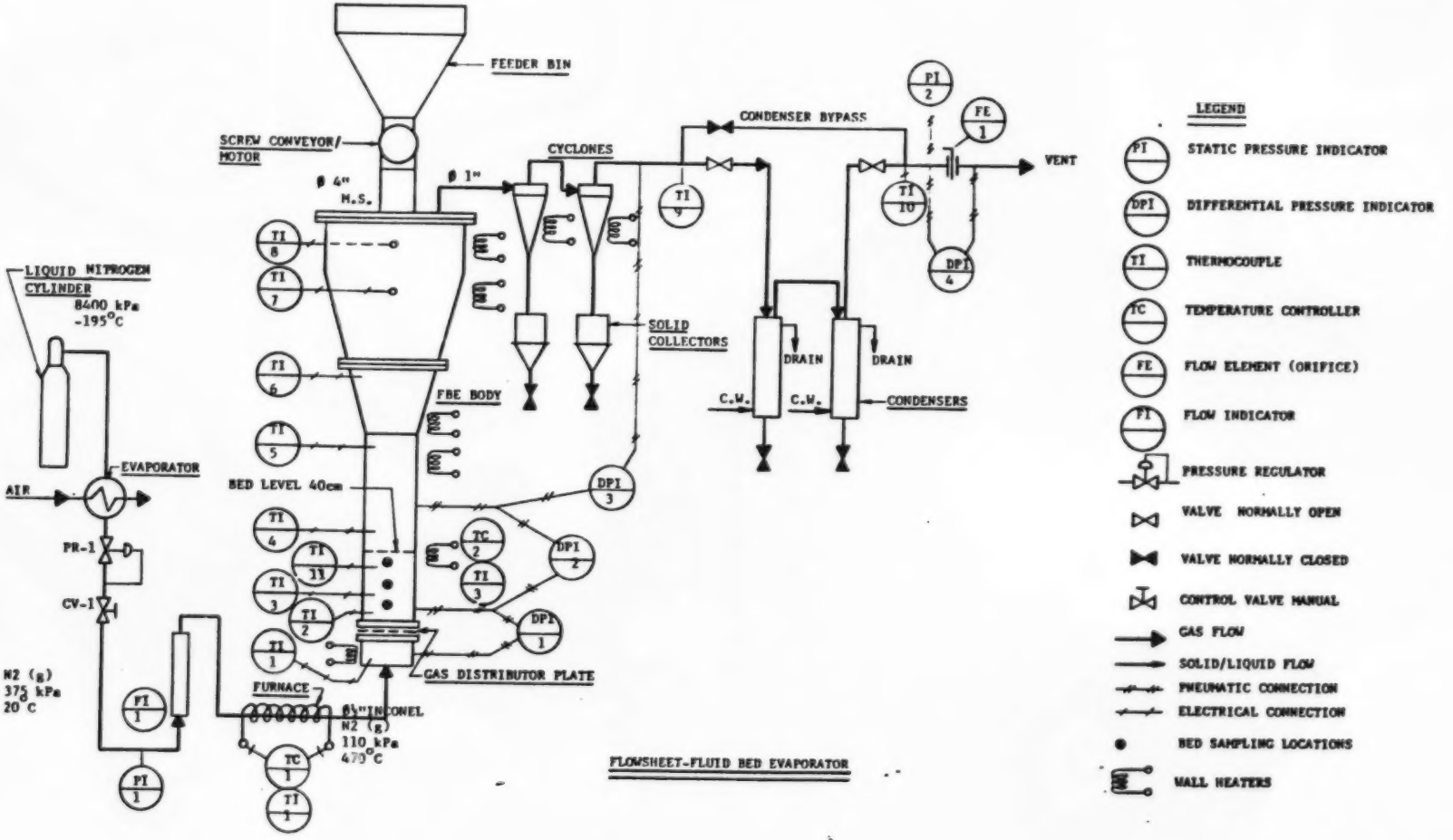
(b) The effects of feed rate, residence time, and feed oil content on the product coal oil content should be further investigated.

(c) Methods of particulate removal from the exhaust gases need some study as indicated by cyclone plugging and the amount of solids removed from the condensers.

(d) The removal and collection of the condensate should be examined in greater detail. This would involve sampling the exhaust stream in order to determine particle-size distribution and the choosing of suitable collection equipment.

APPENDIX A

FBE Flowsheet


FEEDER BIN
SCREW CONVEYOR/ MOTOR
ϕ 4" M.S.
ϕ 1"
CYCLONES
CONDENSER BYPASS
PI 2
FE 1
VENT
TI 9
TI 10
DPI 4
LIQUID NITROGEN CYLINDER
8400 kPa
-195°C
TI 8
TI 7
TI 6
TI 5
FBE BODY
SOLID COLLECTORS
DRAIN
DRAIN
C.W.
C.W.
CONDENSERS
EVAPORATOR
AIR
BED LEVEL 40cm
DPI 3
TI 4
TI 11
TC 2
DPI 2
PR-1
TI 3
CV-1
TI 2
DPI 1
TI 1
GAS DISTRIBUTOR PLATE
N2 (g)
375 kPa
20°C
FI 1
FURNACE
½"INCONEL
N2 (g)
110 kPa
470°C
TC 1
PI 1
TI 1
FLOWSHEET-FLUID BED EVAPORATOR
LEGEND
STATIC PRESSURE INDICATOR
DIFFERENTIAL PRESSURE INDICATOR
THERMOCOUPLE
TEMPERATURE CONTROLLER
FLOW ELEMENT (ORIFICE)
FLOW INDICATOR
PRESSURE REGULATOR
VALVE NORMALLY OPEN
VALVE NORMALLY CLOSED
CONTROL VALVE MANUAL
GAS FLOW
SOLID/LIQUID FLOW
PNEUMATIC CONNECTION
ELECTRICAL CONNECTION
BED SAMPLING LOCATIONS
WALL HEATERS

APPENDIX B

Bed Product Collector

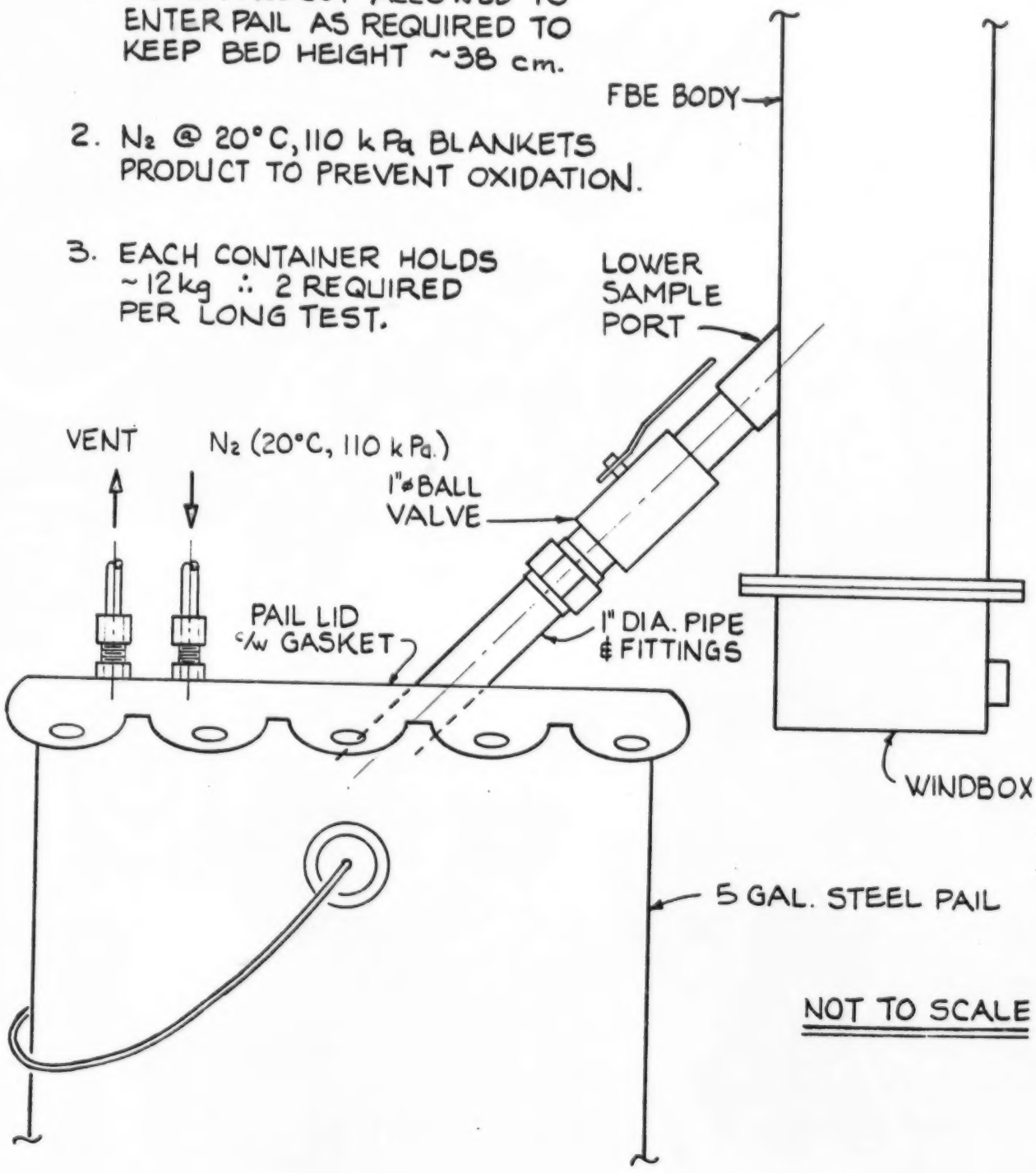
and

Condenser Assembly

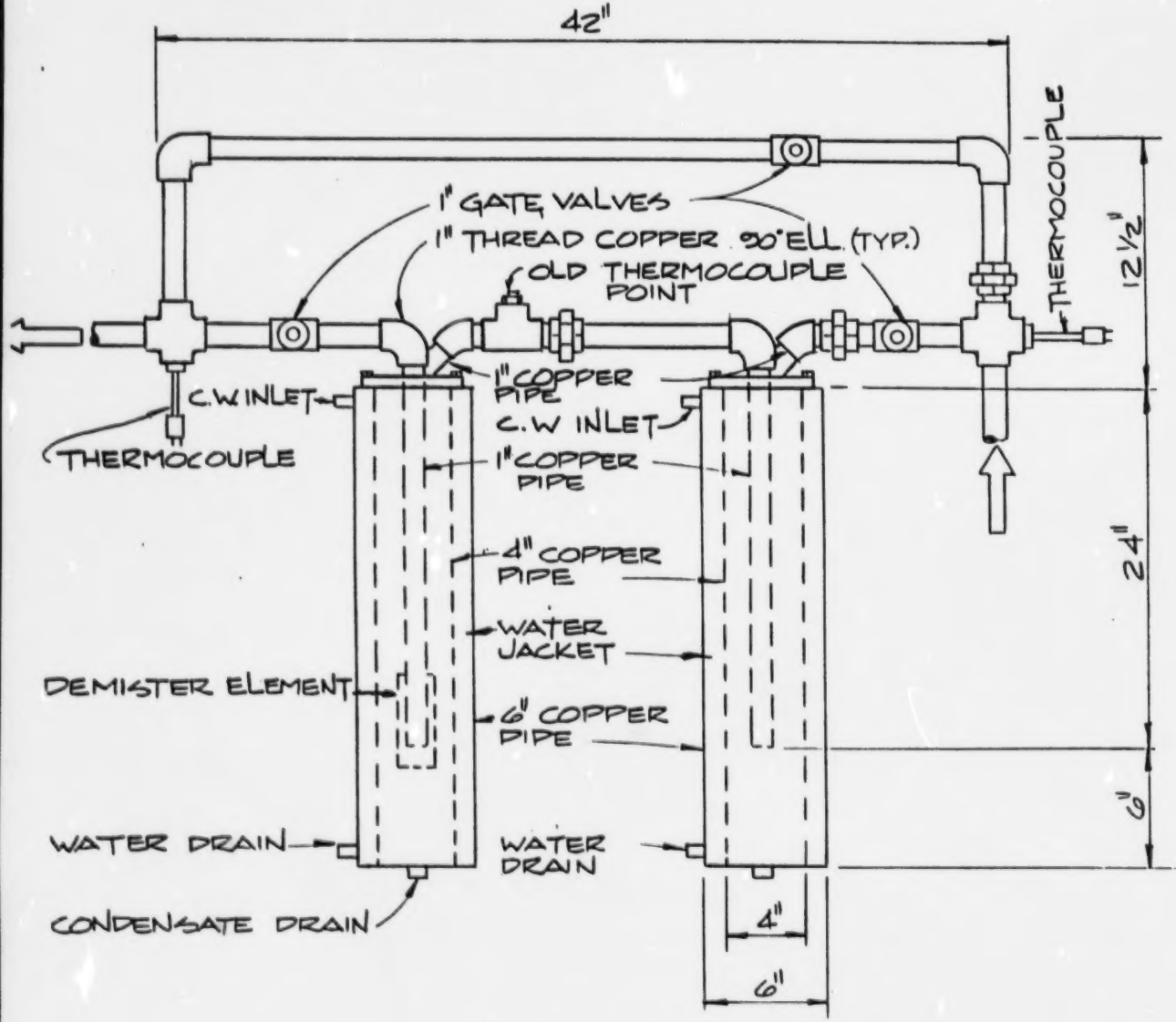
1. COAL PRODUCT ALLOWED TO ENTER PAIL AS REQUIRED TO KEEP BED HEIGHT ~38 cm.

2. N_2 @ 20°C, 110 kPa BLANKETS PRODUCT TO PREVENT OXIDATION.

3. EACH CONTAINER HOLDS ~12kg ∴ 2 REQUIRED PER LONG TEST.



NOT TO SCALE

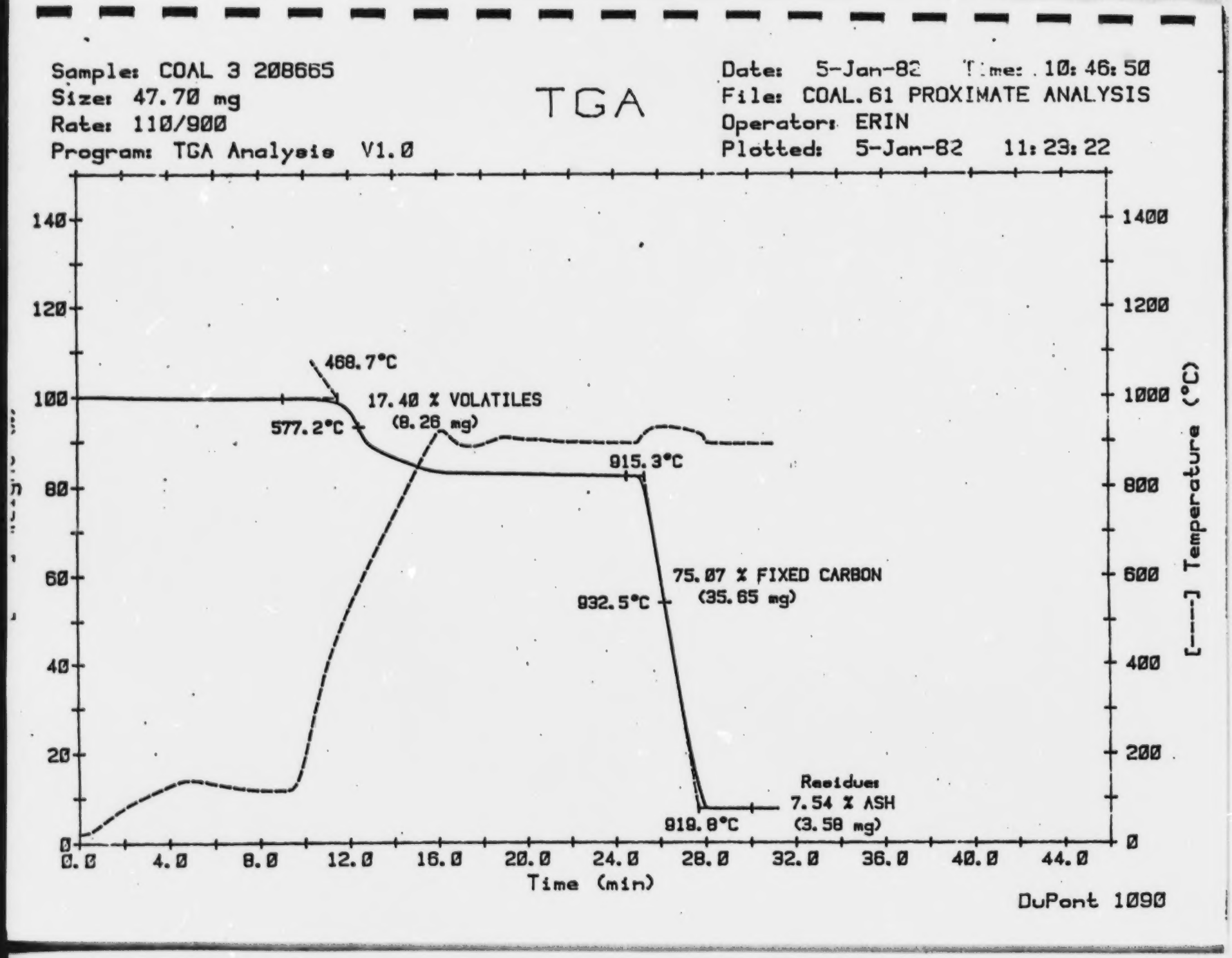


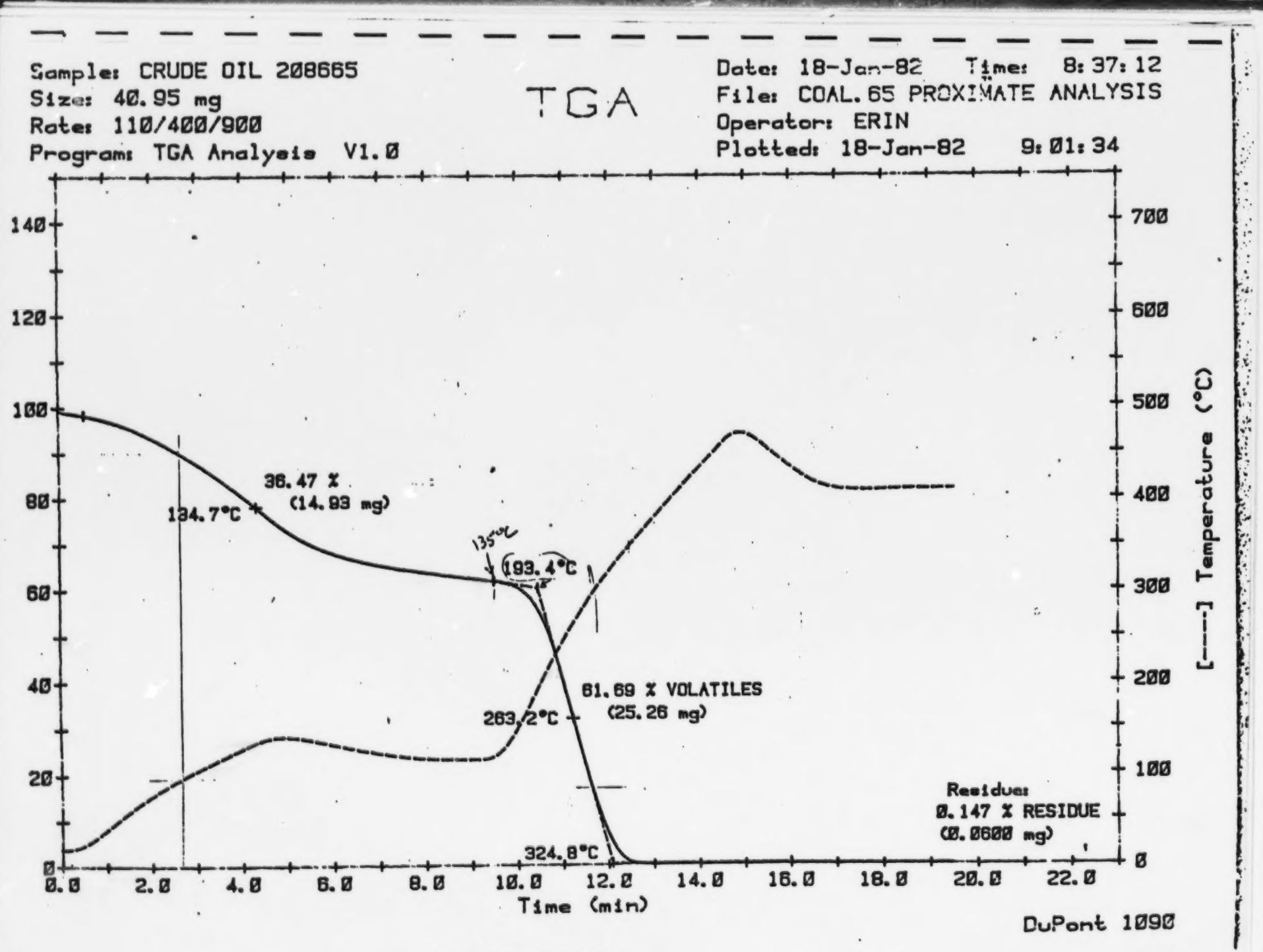
NOTE

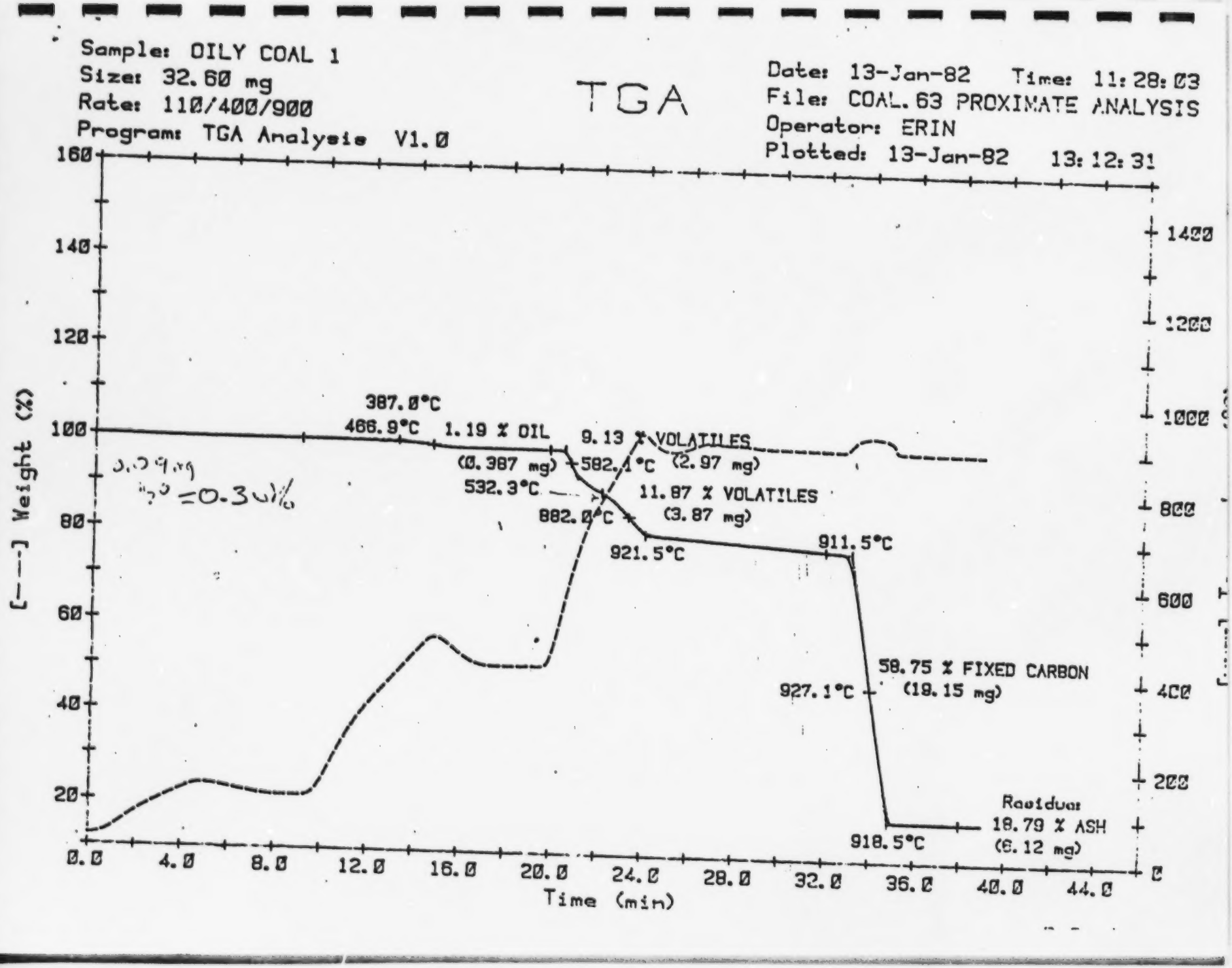
1. ALL PIPE IS 1" BLACK IRON OR STEEL EXCEPT C.W. INLETS & OUTLETS AS INDICATED.
2. TOP PLATE OF CONDENSERS ARE REMOVABLE WITH 8 SELF-TAPPING METAL SCREWS

APPENDIX C

Examples of TGA Plots


Sample: COAL 3 208665
Size: 47.70 mg
Rate: 110/900
Program: TGA Analysis V1.0
TGA
Date: 5-Jan-82 Time: 10:46:50
File: COAL.61 PROXIMATE ANALYSIS
Operator: ERIN
Plotted: 5-Jan-82 11:23:22
468.7°C
17.40 % VOLATILES
(8.26 mg)
577.2°C
915.3°C
75.07 % FIXED CARBON
(35.65 mg)
932.5°C
Residue:
7.54 % ASH
(3.58 mg)
919.8°C
140
120
100
80
60
40
20
0
1400
1200
1000
800
600
400
200
0
[----] Temperature (°C)
0.0
4.0
8.0
12.0
16.0
20.0
24.0
28.0
32.0
36.0
40.0
44.0
Time (min)
DuPont 1090

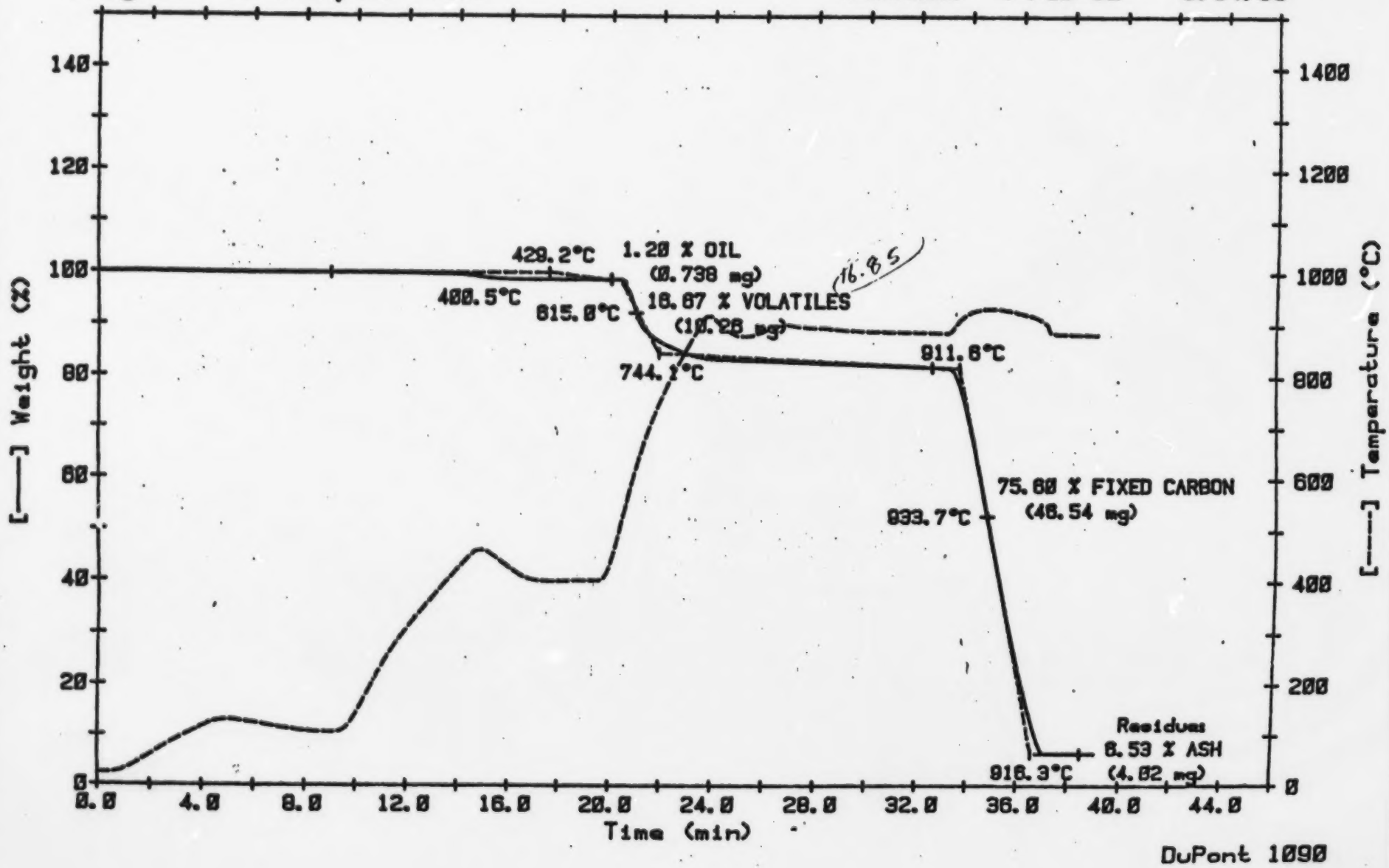

Sample: CRUDE OIL 208665
Size: 40.95 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 18-Jan-82 Time: 8:37:12
File: COAL.65 PROXIMATE ANALYSIS
Operator: ERIN
Plotted: 18-Jan-82 9:01:34
36.47 %
(14.93 mg)
134.7°C
135°C
193.4°C
61.69 % VOLATILES
(25.26 mg)
263.2°C
Residue:
0.147 % RESIDUE
(0.0600 mg)
324.8°C
Time (min)
[----] Temperature (°C)
DuPont 1090


Sample: OILY COAL 1
Size: 32.60 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 13-Jan-82 Time: 11:28:03
File: COAL.63 PROXIMATE ANALYSIS
Operator: ERIN
Plotted: 13-Jan-82 13:12:31
[---] Weight (%)
160
140
120
100
80
60
40
20
1400
1200
1000
800
600
400
200
0
0.0
4.0
8.0
12.0
16.0
20.0
24.0
28.0
32.0
36.0
40.0
44.0
Time (min)
387.0°C
466.9°C
1.19 % OIL
(0.387 mg)
582.1°C
9.13 % VOLATILES
(2.97 mg)
532.3°C
882.0°C
11.87 % VOLATILES
(3.87 mg)
921.5°C
911.5°C
927.1°C
58.75 % FIXED CARBON
(19.15 mg)
Residue
18.79 % ASH
(6.12 mg)
918.5°C
3.09 mg
=0.3 wt%

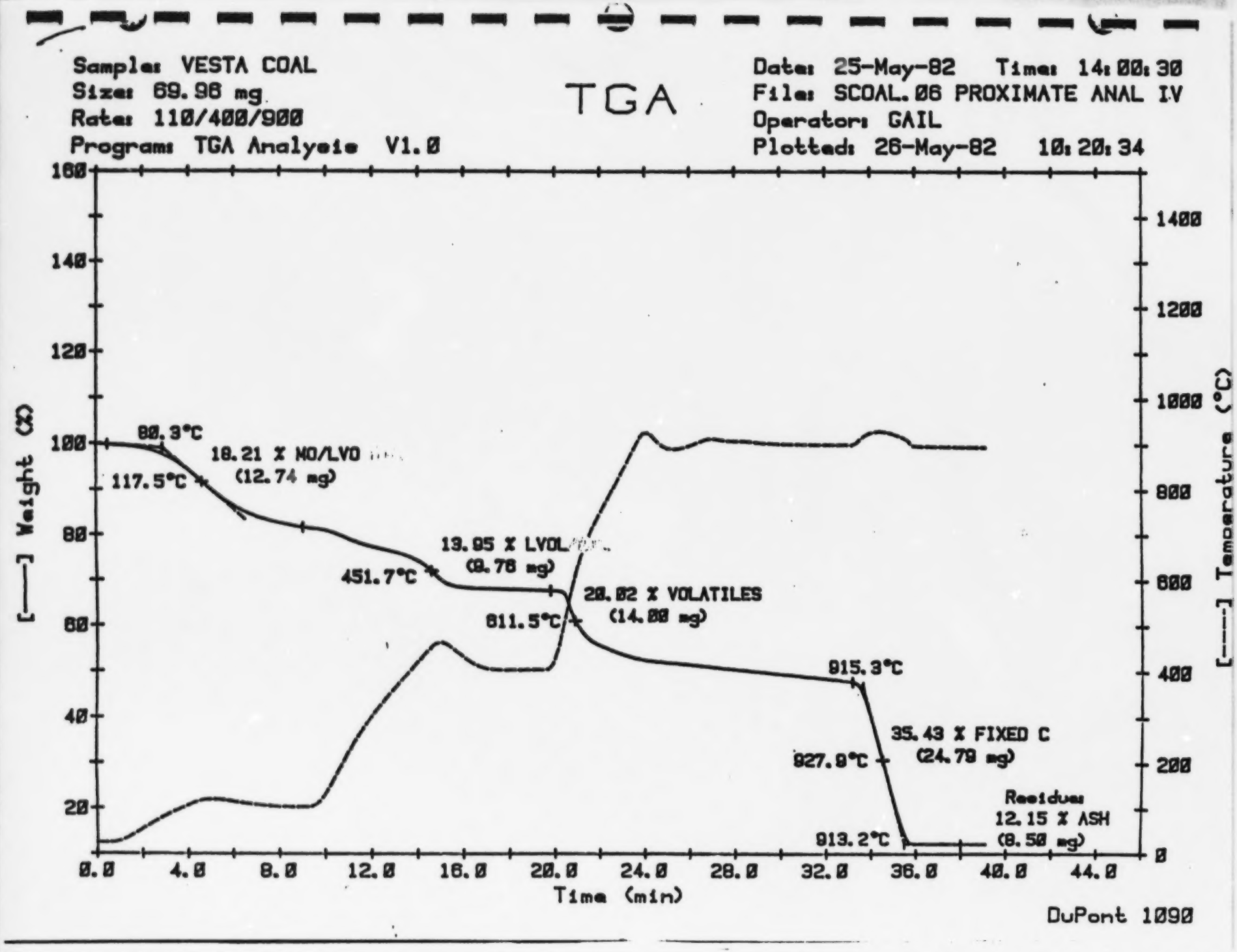
Sample: BC15 (Illinois Coal Run #1)
Size: 61.64 mg
Rate: 110/400/900
Program: TGA Analysis V1.0

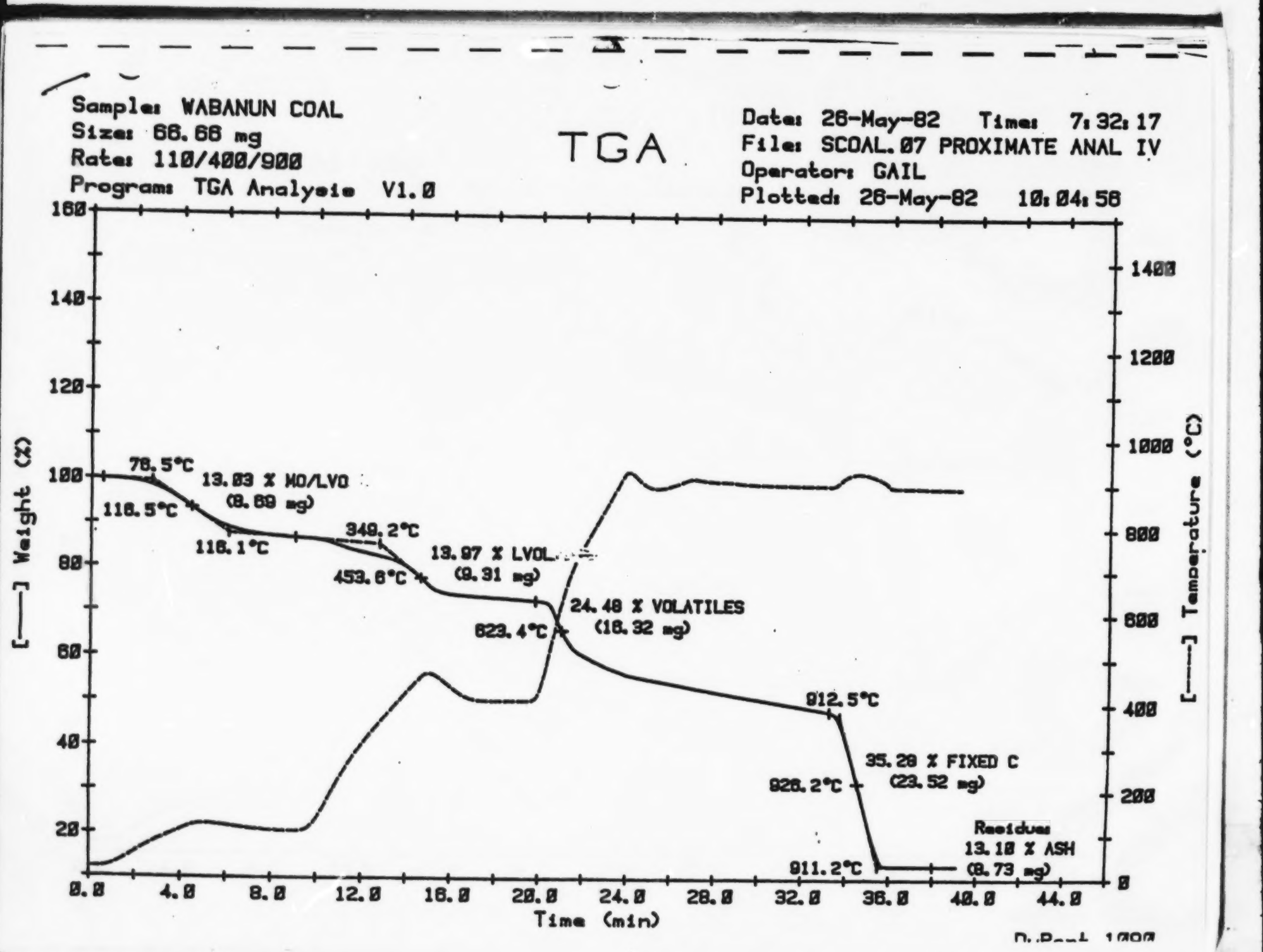
TGA

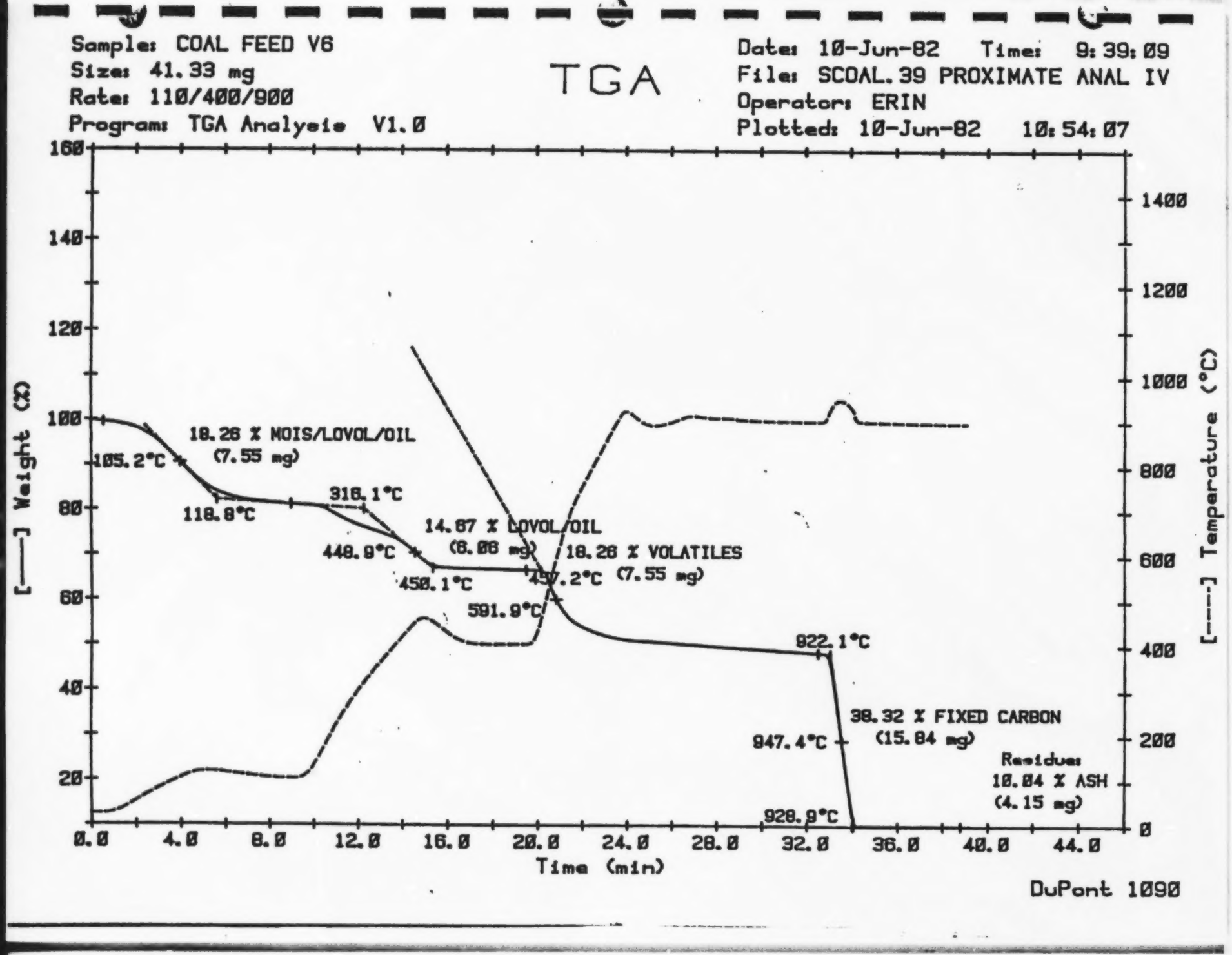
Date: 4-Feb-82 Time: 8:17:13
File: ICOAL.04 MISC TESTING V
Operator: ERIN
Plotted: 4-Feb-82 9:14:35

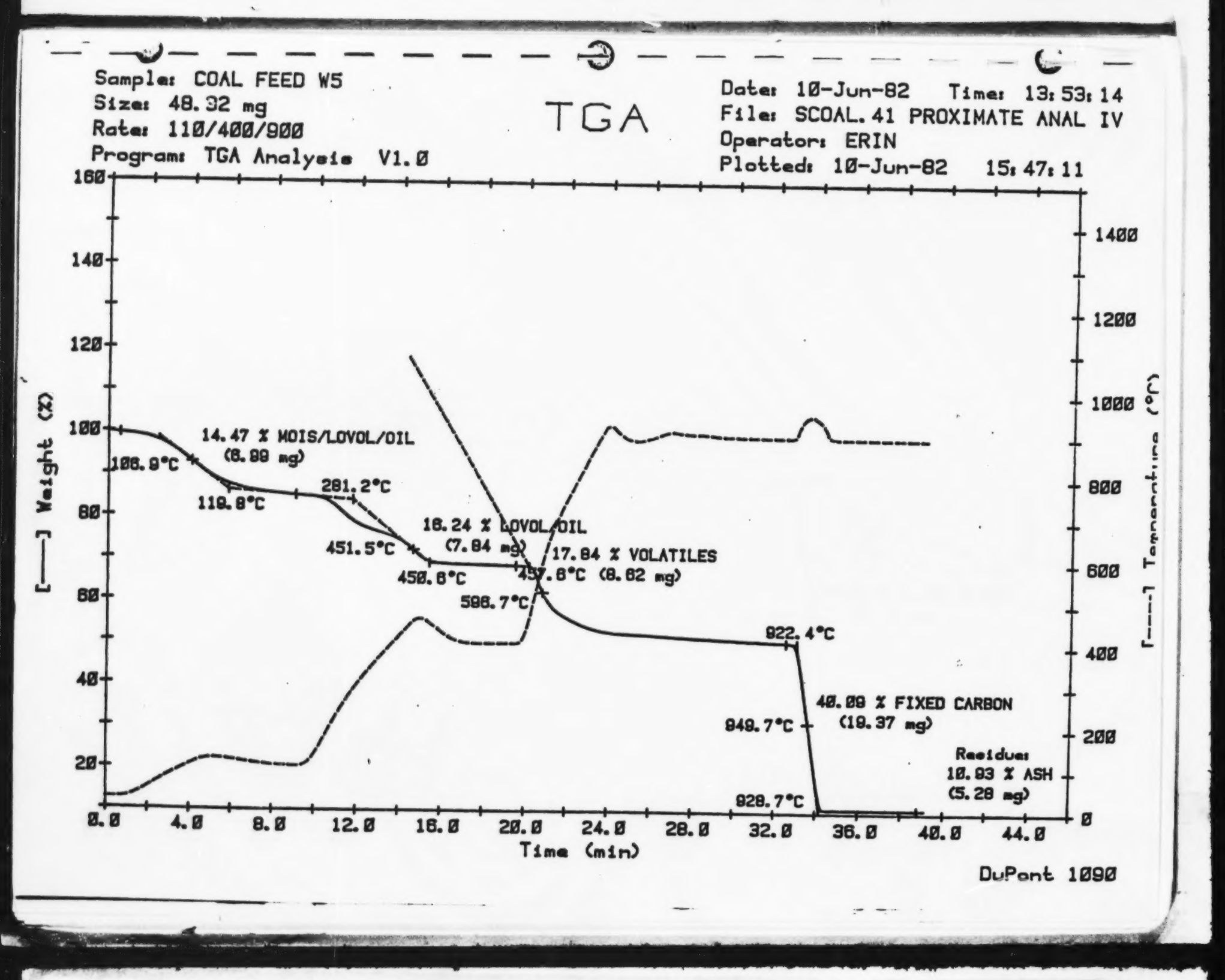


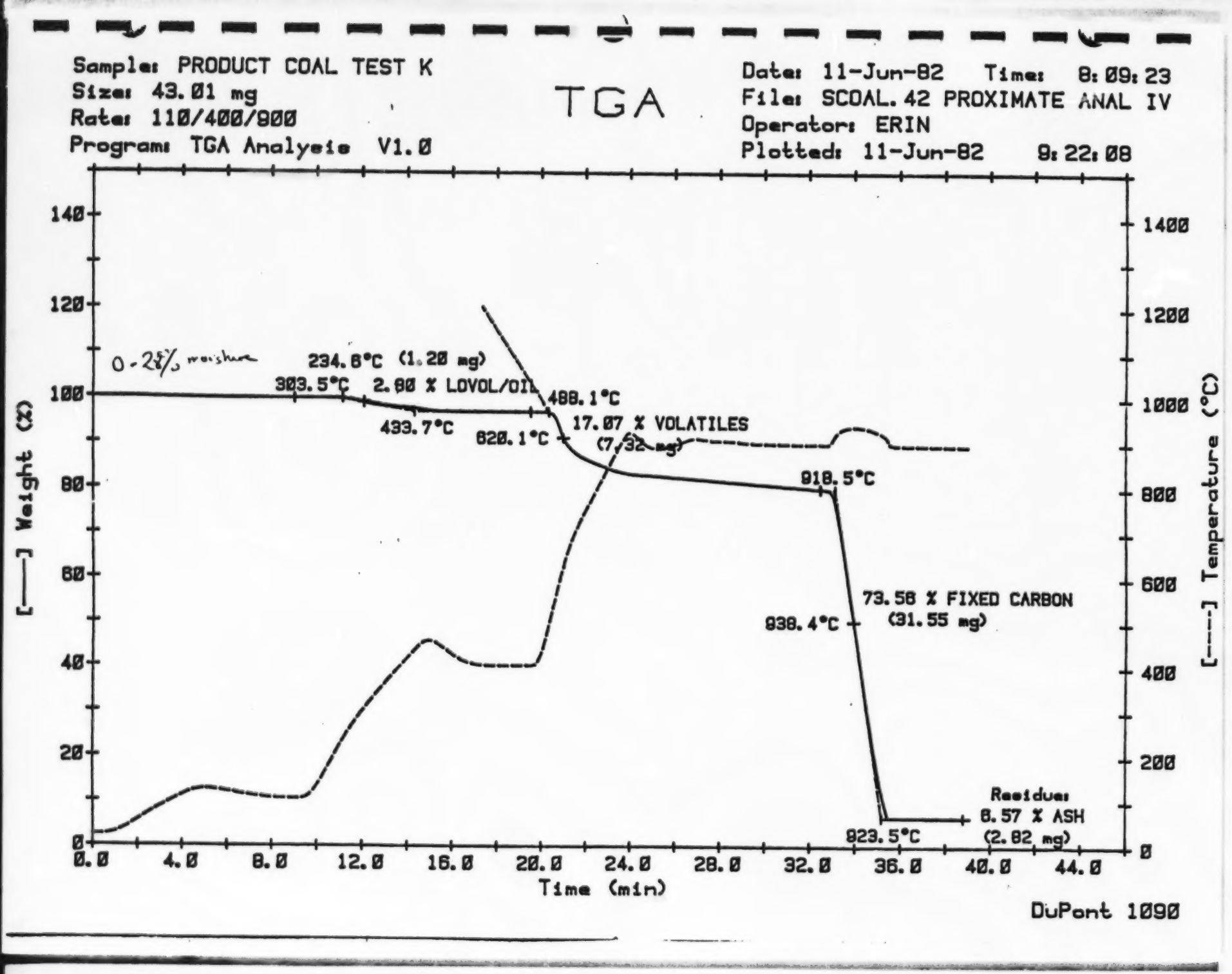
DuPont 1090

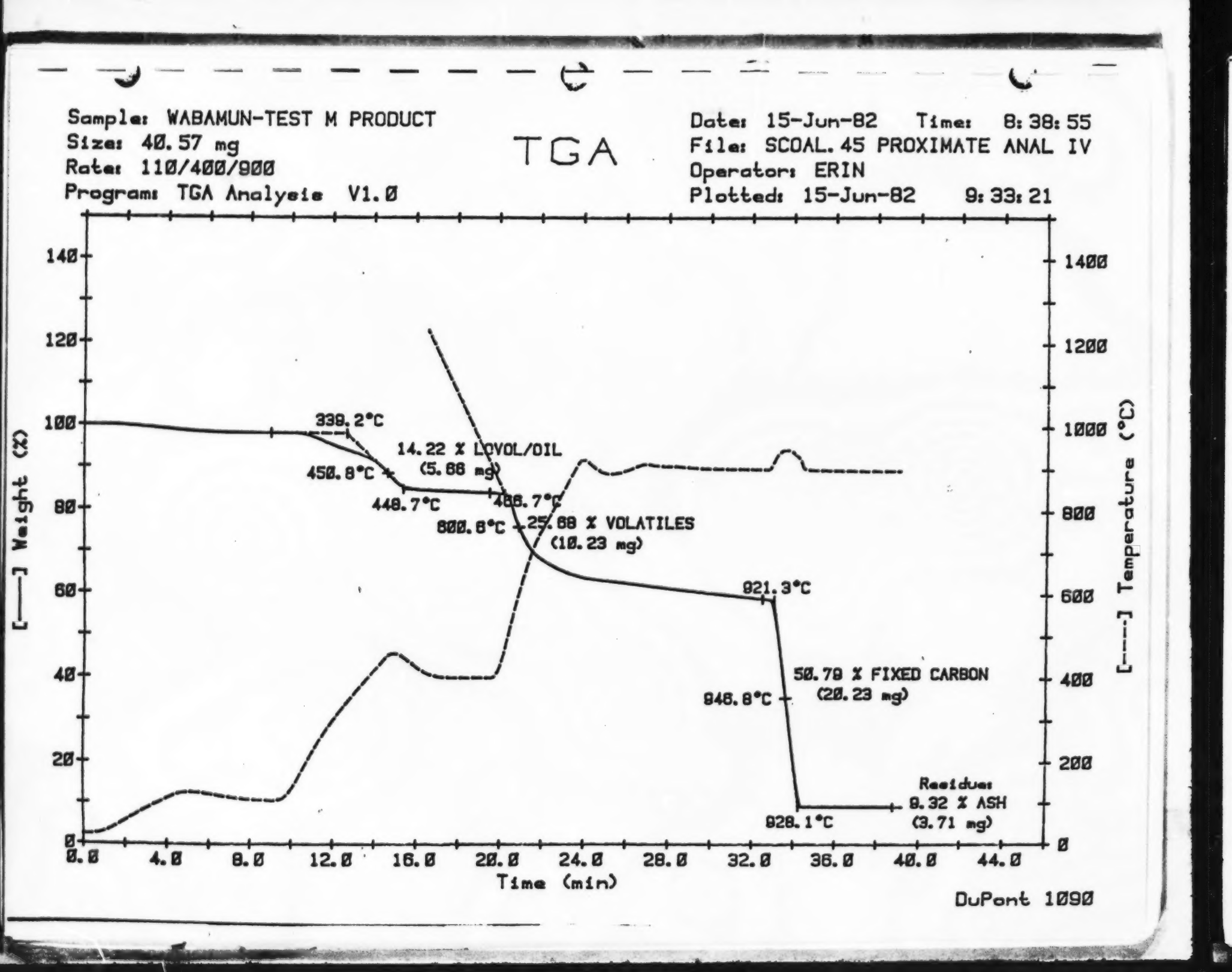

Sample: VESTA COAL
Size: 69.96 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 25-May-82 Time: 14:00:30
File: SCOAL.06 PROXIMATE ANAL IV
Operator: GAIL
Plotted: 26-May-82 10:20:34
[——] Weight (%)
[----] Temperature (°C)
Time (min)
80.3°C
117.5°C
18.21 % MO/LVO
(12.74 mg)
451.7°C
13.95 % LVOL
(9.76 mg)
811.5°C
28.02 % VOLATILES
(14.00 mg)
915.3°C
927.9°C
35.43 % FIXED C
(24.79 mg)
913.2°C
Residue
12.15 % ASH
(8.50 mg)
DuPont 1090


Sample: WABANUN COAL
Size: 66.66 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 26-May-82 Time: 7:32:17
File: SCOAL.07 PROXIMATE ANAL IV
Operator: GAIL
Plotted: 26-May-82 10:04:58
78.5°C
13.03 % MO/LVO
(8.69 mg)
116.5°C
116.1°C
349.2°C
453.6°C
13.97 % LVOL
(9.31 mg)
24.48 % VOLATILES
(16.32 mg)
823.4°C
912.5°C
35.28 % FIXED C
(23.52 mg)
926.2°C
Residue
13.18 % ASH
(8.73 mg)
911.2°C
[——] Weight (%)
[——] Temperature (°C)
Time (min)


Sample: COAL FEED V6
Size: 41.33 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 10-Jun-82 Time: 9:39:09
File: SCOAL.39 PROXIMATE ANAL IV
Operator: ERIN
Plotted: 10-Jun-82 10:54:07
18.26 % MOIS/LOVOL/OIL
(7.55 mg)
185.2°C
118.8°C
316.1°C
448.9°C
14.67 % LOVOL/OIL
(6.06 mg)
450.1°C
18.26 % VOLATILES
457.2°C (7.55 mg)
591.9°C
922.1°C
38.32 % FIXED CARBON
(15.84 mg)
947.4°C
Residue:
10.04 % ASH
(4.15 mg)
928.9°C
[——] Weight (%)
[----] Temperature (°C)
Time (min)
DuPont 1090

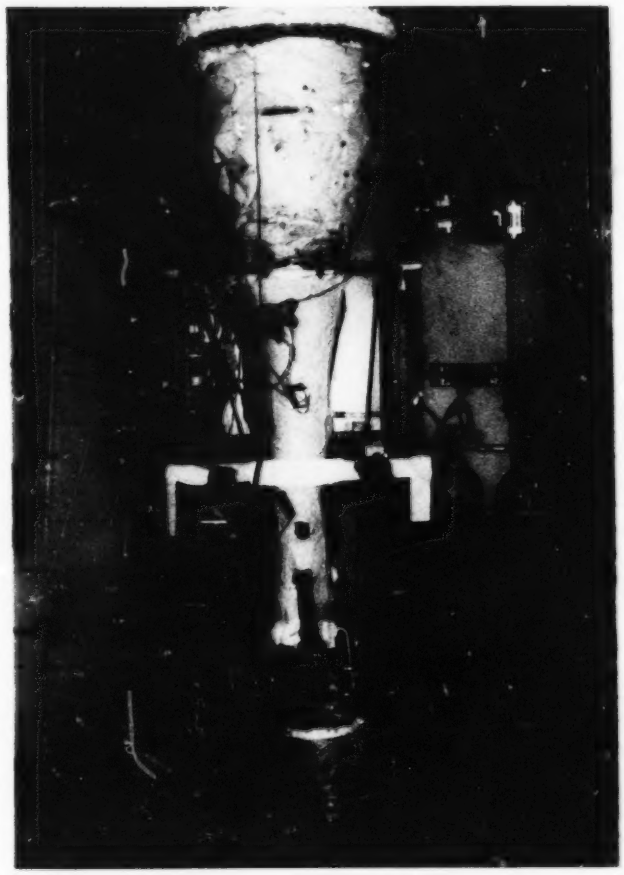

Sample: COAL FEED W5
Size: 48.32 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 10-Jun-82 Time: 13:53:14
File: SCOAL.41 PROXIMATE ANAL IV
Operator: ERIN
Plotted: 10-Jun-82 15:47:11
[——] Weight (%)
[----] Temperature (°C)
Time (min)
14.47 % MOIS/LOVOL/OIL
(8.89 mg)
106.9°C
119.8°C
281.2°C
451.5°C
18.24 % LOVOL OIL
(7.84 mg)
450.6°C
17.84 % VOLATILES
457.6°C (8.62 mg)
598.7°C
922.4°C
40.09 % FIXED CARBON
(19.37 mg)
948.7°C
928.7°C
Residue:
10.93 % ASH
(5.28 mg)
DuPont 1090


Sample: PRODUCT COAL TEST K
Size: 43.01 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 11-Jun-82 Time: 8:09:23
File: SCOAL.42 PROXIMATE ANAL IV
Operator: ERIN
Plotted: 11-Jun-82 9:22:08
0-2% moisture
234.6°C (1.20 mg)
303.5°C 2.80 % LOVOL/OIL
433.7°C
488.1°C
620.1°C
17.07 % VOLATILES
(7.32 mg)
918.5°C
938.4°C
73.58 % FIXED CARBON
(31.55 mg)
Residue:
8.57 % ASH
(2.82 mg)
923.5°C
[——] Weight (%)
[----] Temperature (°C)
Time (min)
0.0 4.0 8.0 12.0 16.0 20.0 24.0 28.0 32.0 36.0 40.0 44.0
0 20 40 60 80 100 120 140
0 200 400 600 800 1000 1200 1400
DuPont 1090


Sample: WABAMUN-TEST M PRODUCT
Size: 40.57 mg
Rate: 110/400/900
Program: TGA Analysis V1.0
TGA
Date: 15-Jun-82 Time: 8:38:55
File: SCOAL.45 PROXIMATE ANAL IV
Operator: ERIN
Plotted: 15-Jun-82 9:33:21
339.2°C
14.22 % LOVOL/OIL
(5.66 mg)
450.8°C
448.7°C
486.7°C
800.8°C
25.68 % VOLATILES
(10.23 mg)
921.3°C
50.79 % FIXED CARBON
(20.23 mg)
848.8°C
Residue:
9.32 % ASH
(3.71 mg)
928.1°C
[——] Weight (%)
[----] Temperature (°C)
Time (min)
DuPont 1090

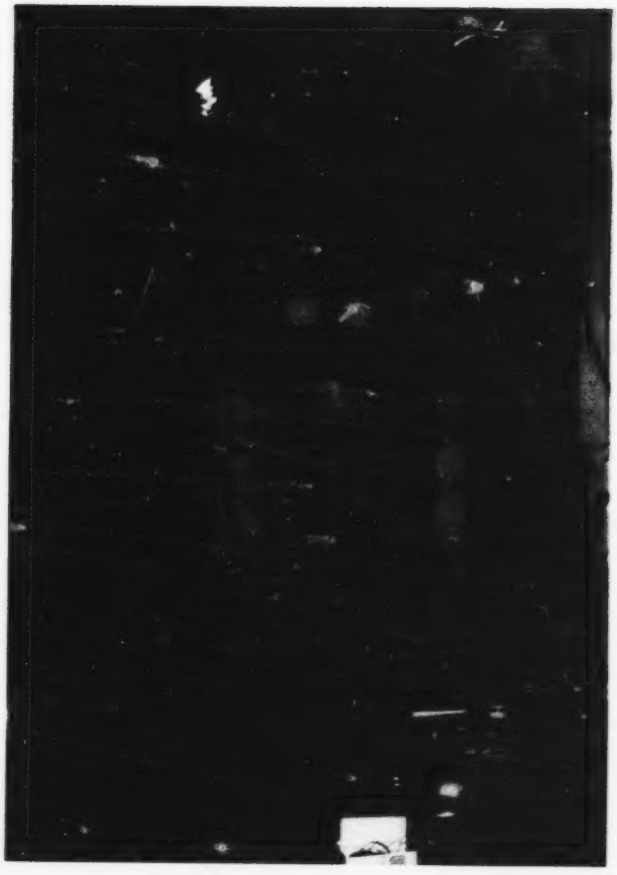
APPENDIX D

Photographs



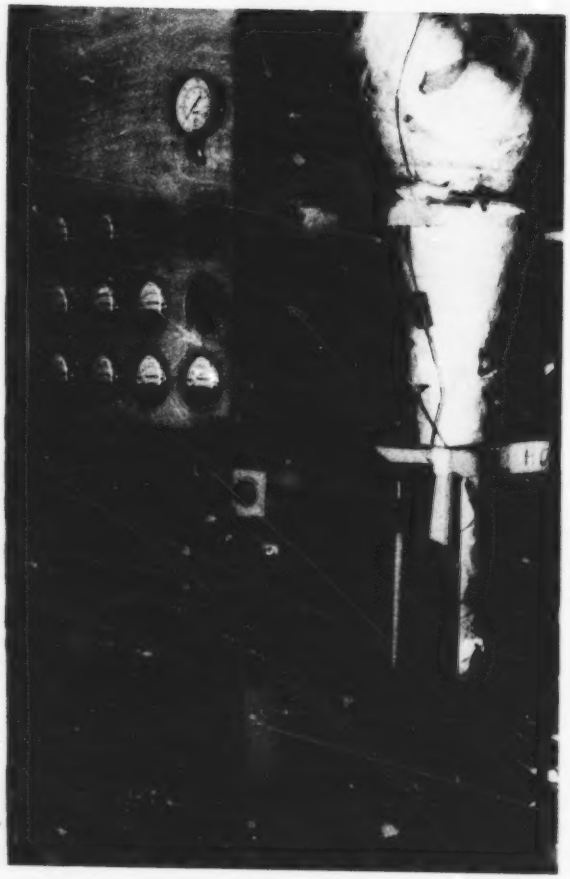
1

FRONT VIEW OF LOWER FBE ASSEMBLY



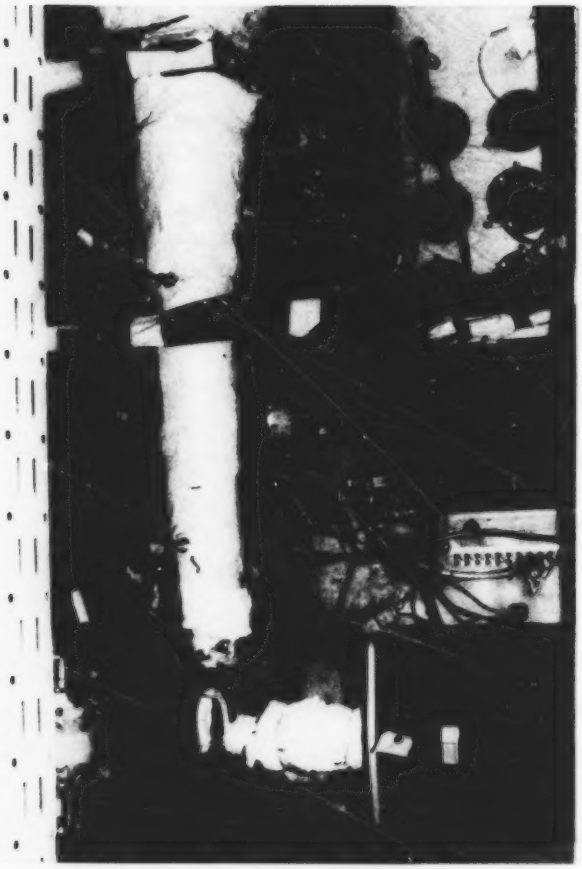
2.

FBE ASSEMBLY INCLUDING MASS FEEDER AND EXHAUST



3.

VIEW OF CONTROL PANEL



4.

SIDE VIEW OF FBE ASSEMBLY

SHOWING FURNACE AND WINDBOX

6.

VIEW OF FEEDER AND EXHAUST CONNECTIONS

6.

CONDENSER ASSEMBLY